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September 5, 2013

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EPA CONTRACT NUMBER EP-W-10-033
TASK ORDER NUMBER 2021
QA SUPPORT FOR RI/FS AT THE LIBBY ASBESTOS SITE OU3

Dear Ms. Zinner:

Enclosed please find the finalized Annual QA/QC Summary Report (2007-2012). This report is a deliverable under Task 9 of Task Order 2021.

If you have any questions, please feel free to contact me.

Sincerely,

Timothy L. Vonnahme

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ANNUAL QA/QC SUMMARY REPORT (2007-2012) (FINAL)

FOR TASK ORDER 2021 QUALITY ASSURANCE (QA) SUPPORT FOR REMEDIAL INVESTIGATION/FEASIBILITY STUDY (RI/FS) AT SITE OU3

Prepared by:

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September 5, 2013

QATS Contract Number: EP-W-10-033

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OFFICE OF SUPERFUND REMEDIATION AND TECHNOLOGY INNOVATION
U. S. ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

Table of Contents

Acronym List

<	Less Than
≥	Greater Than or Equal To
%	Percent
AHERA	Asbestos Hazard Emergency Response Act
AOC	Administrative Order on Consent
ASTM	American Society for Testing and Materials
COC	Chain-Of-Custody
CSF	Close Support Facility
EDD	Electronic Data Deliverable
EPA	Environmental Protection Agency
ESAT	Environmental Services Assistance Team
f/cc	Fibers per Cubic Centimeter
f/mm2	Fibers per Square Millimeter
FB	Field Blank
ISO	International Organization for Standardization
ISSI	ISSI Consulting Group, Inc.
LA	Libby Amphibole
LDC	Laboratory Duplicate Cross-check
LDS	Laboratory Duplicate Self-check
MAS	Material Analytical Services, LLC
MFL	Million Fibers per Liter
ND	Non-Detect
NVLAP	National Voluntary Laboratory Accreditation Program
OU3	Libby Operable Unit 3
PCM	Phase Contrast Microscopy
PCME	PCM-Equivalent
PES	Performance Evaluation Sample
PLM	Polarized Light Microscopy
PLM-GRAV	Polarized Light Microscopy Gravimetric
PLM-VE	Polarized Light Microscopy-Visual Area Estimation
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QATS	Quality Assurance Technical Support
RD	Recount Different
RI/FS	Remedial Investigation/Feasibility Study
RS	Recount Same
SAED	Selected Area Electron Diffraction
SAP	Sampling and Analysis Plan
s/cc	Structures per Cubic Centimeter
SOP	Standard Operating Procedure
SPF	Soil Preparation Facility
SRM	Solid Reference Material
TEM	Transmission Electron Microscopy
VA	Verified Analysis

1.0 Introduction

1.1 Purpose of this Report

This Annual Summary Report is a requirement of Task 9 of Task Order 2-021, *Quality Assurance/Quality Control (QA/QC) Support for Remedial Investigation/Feasibility Study (RI/FS) at the Libby Asbestos Site OU3*, issued to Shaw Environmental, Inc. (a CB&I Company) on November 14, 2012 by the U.S. Environmental Protection Agency (EPA) Region 8. The purpose of this report is to provide an updated summary of the QA activities conducted on analyses from the OU3 site from 2007 through 2012. Operable Unit 3 (OU3) is one of eight Operable Units designated by EPA for the Libby RI/FS, which encompasses the mine property itself and areas impacted by releases from the mine, such as creeks, the Kootenai River, settling ponds, the surrounding forest, and Rainy Creek Road. The Libby RI/FS at OU3 is being conducted through an Administrative Order on Consent (AOC) entered into by EPA with respondents W.R. Grace & Co. and Kootenai Development Corporation (KDC).

1.2 Report Outline

The elements provided in this report are as defined in the Task Order, and include:

- Data Management QA
- QC Data Evaluated
- Asbestos Data Validation
- Laboratory Audits
- Laboratory Mentoring Program
- Development and Review of Standard Operating Procedures (SOPs) and Other Quality Documents
- Conclusions and Recommendations

The primary contaminant at OU3 is Libby Amphibole (LA) which is a form of asbestos present in the vermiculite that had been mined at the site from 1919 to 1990. The QC data summarized in the report includes data from the preparation and analysis of LA from OU3. The data evaluated for some of the above elements span multiple years. For example, the QC data evaluated includes data collected from 2007 through 2012; asbestos data validation of samples collected from 2007 through 2012; and laboratory audits performed on OU3 support laboratories in 2008, 2011, and 2012. Where possible, QA/QC trends (e.g., year-over-year performance in on-site laboratory audits, data validation results) are discussed in the report.

2.0 Data Management QA

This section will be completed by CDM Smith.

3.0 QC Data Evaluated

The QC data described in this section were collected from the OU3 site between 2007 and 2012, and are categorized in the tables by investigation phases performed to support specific types of studies. The approximate dates and titles of the ten investigation phases performed at the Libby OU3 Superfund Site during this time period include:

Phase I - Sampling and Analysis Plan, September 2007
Phase II, Part A - Surface Water and Sediment, May 2008
Phase II, Part B - Air and Groundwater, July 2008
Phase II, Part C - Ecological Data, September 2008
Phase III - Sampling and Analysis Plan, May 2009
Phase IV, Part A - Data to Support Human Health Risk Assessment, June 2010
Phase IV, Part B - Surface Water Study, April 2011
Phase V, Part A - Surface Water, Sediment, and Activity-Based Sampling, October 2012
Phase V, Part B - Ecological Investigations, May 2012
Phase CL - Commercial Logging, August 2012

This section summarizes the result summaries by the following categories:

- Field Quality Control
- Preparation Quality Control
- Laboratory Analysis Quality Control
- Performance Evaluation Sample (PES) Results

3.1 Field Quality Control

Field-based QC samples are samples collected in the field and submitted to the laboratory along with the regular field samples for analysis. Four types of field QC samples were collected from OU3 between 2007 and 2012:

- Field Blank
- Rinsate Blank
- Lot Blank
- Field Duplicate/Split

Field QC samples were collected with samples to be analyzed by PLM-Visual Estimation (VE), PLM-Gravimetric (GRAV), and TEM. Field QC samples for PLM-VE analyses include field duplicates of forest soil, mine waste, and sediment samples collected for Phase I in 2007, and field duplicates of sediment and soil samples collected for Phases II and V in 2008 and 2012. A total of 31 PLM-VE field duplicate samples of 437 field samples were collected, which represent an overall field QC to sample ratio of 7.1%. **Table 1** provides a summary of the PLM-VE field QC collected by Phase.

Table 1 - PLM-VE Field QC

			Field Duplicates	
Investigation	**Media**	**Field Samples**	**No. of Analyses**	**%**
Phase I	Forest Soil	82	8	9.8%
	Mine Waste	42	4	9.5%
	Sediment	27	3	11.1%
	Soil	23	0	0.0%
Phase IIA	Sediment	128	12	9.4%
	Soil	48	0	0.0%
Phase IIC	Sediment	13	1	7.7%
	Soil	3	0	0.0%
Phase VA	Sediment	6	1	16.7%

Table 1 - PLM-VE Field QC

			Field Duplicates	
Investigation	**Media**	**Field Samples**	**No. of Analyses**	**%**
Phase VB	Sediment	65	2	3.1%
	Totals	**437**	**31**	**7.1%**

Field QC samples for PLM-GRAV analyses include field duplicates of forest soil, mine waste, and sediment samples collected for Phase I in 2007, and to sediment samples from Phases II and V in 2008 and 2012. PLM-GRAV field QC includes 17 field duplicate samples for 194 field samples collected, which represents 8.8% of the total. **Table 2** provides a summary of the PLM Field QC collected by investigation.

Table 2 - PLM-GRAV Field QC

			Field Duplicates	
Investigation	**Media**	**Field Samples**	**No. of Analyses**	**%**
Phase I	Forest Soil	73	6	8.2%
	Mine waste	42	4	9.5%
	Sediment	19	2	10.5%
	Soil	12	0	0.0%
Phase IIA	Sediment	24	3	12.5%
	Soil	3	0	0.0%
Phase IIC	Sediment	9	0	0.0%
Phase VB	Sediment	12	2	16.7%
	Totals	**194**	**17**	**8.8%**

Field QC samples for TEM analyses were applied to air, duff, water, and tree bark samples analyzed from 2007 through 2012 for investigation phases I-V. TEM field QC samples include field duplicate samples, equipment rinsate blanks, field blanks, sample splits, and lot blanks. Of the 1,551 total samples collected from the OU3 site for TEM analysis during this period, 193 various field QC samples were applied, which represents 12.4% of the total. **Table 3** shows the type, number, and frequency of TEM Field QC samples by investigation.

Table 3 - TEM Field QC, Phases I-CL

			Field Duplicates		Rinsate Blanks		Field Blanks		Lot Blanks		Sample Splits	
Investigation	**Media**	**Field Samples**	**No. of Analyses**	**%**	**No. of Analyses**	**%**	**No. of Analyses**	**%**	**No. of Analyses**	**%**	**No. of Analyses**	**%**
Phase I	Air	49	4	8.2%	---	---	8	16.3%	5	10.2%	---	---
	Duff	81	8	9.9%	---	---	---	---	---	---	0	0.0%
	Surface Water	30	3	10.0%	---	---	3	10.0%	---	---	0	0.0%
	Tree Bark	82	8	9.8%	---	---	---	---	---	---	0	0.0%
Phase IIA	Surface Water	290	19	6.6%	---	---	23	7.9%	---	---	2	0.7%
Phase IIB	Air	82	8	9.8%	---	---	8	9.8%	1	1.2%	---	---
	Groundwater	28	1	3.6%	5	17.9%	0	0.0%	---	---	2	7.1%
Phase IIC	Surface Water	4	1	25.0%	---	---	1	25.0%	---	---	0	0.0%
Phase III	Air (ABS)	241	---	--	---	---	7	2.9%	4	1.7%	---	---
Phase IVA	Air (ABS)	262	---	---	---	---	6	2.3%	4	1.5%	---	---
	Tree Bark	5	---	----	---	----	0	0.0%	----	---	0	0.0%
Phase IVB	Surface Water	104	11	10.6%	---	---	11	10.6%	---	---	0	0.0%

Table 3 - TEM Field QC, Phases I-CL

			Field Duplicates		Rinsate Blanks		Field Blanks		Lot Blanks		Sample Splits	
Investigation	**Media**	**Field Samples**	**No. of Analyses**	**%**	**No. of Analyses**	**%**	**No. of Analyses**	**%**	**No. of Analyses**	**%**	**No. of Analyses**	**%**
Phase VA	Air (ABS)	3	--	---	---	----	1	33.3%	0	0.0%	---	---
	Surface Water	88	10	11.4%	---	---	6	6.8%	---	---	0	0.0%
Phase VB	Pore Water	49	2	4.1%	---	---	1	2.0%	---	---	0	0.0%
	Surface Water	124	7	5.6%	---	---	8	6.5%	---	---	0	0.0%
CL	Air (ABS)	17	---	---	---	---	3	17.6%	0	0.0%	---	---
	Duff	6	1	16.7%	---	---	---	---	---	---	0	0.0%
	Tree Bark	6	1	16.7%	---	---	---	---	---	---	0	0.0%
	Totals	**1551**	**84**	**5.4%**	**5**	**0.3%**	**86**	**5.5%**	**14**	**0.9%**	**4**	**0.3%**

3.1.1 Field Blanks

A field blank is a sample of the same medium as the associated field samples used to determine if cross-contamination is occurring during sample collection and/or analysis. Field blank collection frequency requirements are specified in the associated Sampling and Analysis Plans (SAPs). Field blanks for OU3 were collected for water and air samples, but not for sediments, tree bark, duff, and other solid media types. With the exception of the following, field blanks were collected at the frequencies specified in the applicable SAP:

- Phase IIA Surface Water - The SAP specifies a collection frequency of 10%; however, field blanks were only collected at a frequency of 7.9%.
- Phase III ABS Air – Field blanks were only collected on 8 of the 10 days samples were collected.
- Phase VB Pore Water - The SAP specifies a collection frequency of 5%; however, field blanks were only collected at a frequency of 2.0%.

As shown in **Table 3** above, a total of 86 field blanks were collected across all investigation phases from 2007 through 2012. There were four field blanks in which at least one LA fiber was observed, as summarized in **Table 4A** below. One LA fiber was observed in each of the Field Blanks P1-00257 and P5-20103, suggesting that there may have been potential contamination introduced during sample collection and/or analysis. Field blank P1-00257 was collected on 10/18/2007; however, there are no field samples associated with this blank. Field blank P5-20103 was collected on 6/4/2012 along with the pore water samples listed in **Table 4B** below, which should be FB-qualified to alert potential data users. The LA contamination reported in Field Blanks P5-10028 and P5-10014, in which 3 LA fibers and 25 LA fibers were observed, respectively, is part of an investigation described in a technical memorandum to EPA on August 30, 2013 (**see Attachment 1**) and appears to be due to possible mislabeling in the field or misidentification by the laboratory. No LA fibers were observed in any of the other 82 field blanks collected.

Table 4A - TEM Field Blanks with Structures Reported

Index ID	Investigation	Date Collected	Analysis	Media	Structures	Concentration (MFL)
P1-00257	Phase I	18-Oct-07	TEM	Surface Water	1	4.98E-02
P5-10028	Phase VA	16-May-12	TEM	Surface Water	3	4.74E-02
P5-10014		09-May-12	TEM	Surface Water	25	9.23E-01
P5-20103	Phase VB	04-Jun-12	TEM	Pore Water	1	4.43E-02

Table 4B – Samples Associated With Contaminated Field Blanks

Index ID	Field QC Type	Media	Sample Date
P5-20094	Field Sample	Surface Water	6/4/2012
P5-20095	Field Sample	Surface Water	6/4/2012
P5-20096	Field Sample	Surface Water	6/4/2012
P5-20097	Field Sample	Pore Water (In)	6/4/2012
P5-20098	Field Sample	Surface Water	6/4/2012
P5-20099	Field Sample	Pore Water (In)	6/4/2012
P5-20100	Field Sample	Surface Water	6/4/2012
P5-20101	Field Sample	Pore Water (In)	6/4/2012
P5-20102	Field Duplicate	Pore Water (In)	6/4/2012

3.1.2 Rinsate Blanks

Rinsate blanks, which were only applied to groundwater in Phase IIB, are used to determine whether the decontamination procedures applied to field equipment are adequate to prevent cross-contamination of samples during sample collection. Rinsate blanks are prepared by rinsing decontaminated field equipment with analyte-free reagent water. Rinsate blanks are typically collected at a rate of one per sampling team per day. If field equipment is not re-used between sampling locations (i.e., dedicated equipment is used or equipment is disposable and decontamination is not necessary), rinsate blanks are not collected.

As shown in **Table 3**, a total of five rinsate blanks for the 28 groundwater samples were collected in Phase IIB. Seven LA fibers were observed in one rinsate blank sample which resulted in a total LA concentration of 0.35 million fibers per liter (MFL) (Note that all fibers from this rinsate blank were less than 10 µm in length). This indicates that the decontamination procedures applied were not effective and that LA may have been introduced into the samples due to cross-contamination. Two groundwater samples (P2-00780 and P2-00781) were collected on the same day with this rinsate blank, with total LA concentrations ranging from non-detect to 0.1 MFL. Due to the contamination in the rinsate blank, the samples collected on the same day were FB-qualified.

With the exception of the following, rinsate blanks were collected at the frequencies specified in the applicable SAP:

- Phase IIB Groundwater – Rinsate blanks were only collected on 4 of the 7 days samples were collected.

3.1.3 Lot Blanks

Before air filter cassettes can be used for asbestos sampling, the lot must be determined to be asbestos-free. Two lot blanks are selected at random from each group of cassettes to be used for collection of air samples. The lot blanks are analyzed for asbestos fibers by the same method used for field sample analysis. If any asbestos fibers are detected on the lot blanks, the entire batch of cassettes is rejected. Only lots of filters with acceptable lot blank results are placed in the general supply area for use by project personnel.

A total of 14 lot blanks for air cassettes were collected. No asbestos structures were observed in any of the lot blanks analyzed. From these results it was concluded that the likelihood of asbestos contamination in air filter cassettes was low, and the cassette lots were subsequently utilized for the air program.

It should be noted that lot blanks have not been collected and analyzed since 2010. The field team was contacted concerning this situation and it is still unknown whether or not the current lot was checked prior to use. Lot blanks should be checked by comparing the lot numbers of those analyzed versus what the field team recorded in the field logbooks or on the field sample data sheet (FSDS).

3.1.4 Field Splits

A field split is a QC sample that is prepared by thoroughly homogenizing a field sample, dividing the homogenized sample into two parts, and analyzing each independently. Field splits provide a measure of the precision of the sample preparation and analysis methods. As shown in **Table 3**, only four field splits (two surface water and two groundwater, collected respectively in Phases IIA and IIB) were performed for the 1,551 TEM field samples that were collected from 2007 through 2012. Neither of these original-duplicate pair field splits were found to be statistically different from each other using the Poisson ratio comparison test based on a 90% confidence interval.

3.1.5 Field Duplicates

A field duplicate is a second sample that is collected at the same location or coordinates and at approximately the same time as the original field sample, using the same collection technique. Field duplicates are used to evaluate variability due to small-scale media heterogeneity, along with analytical precision. Because field duplicate samples are expected to have inherent variability that is random and may be either small or large, there is no quantitative requirement for the agreement of field duplicates. Rather, results are used to determine the magnitude of this variability to evaluate data usability. With the exception of the following, field duplicates were collected at the frequencies specified in the applicable SAP:

- Phase I Air - The SAP specifies a collection frequency of 10%; however, field duplicates were only collected at a frequency of 8.2%.
- Phase IIA Surface water - The SAP specifies a collection frequency of 10%; however, field duplicates were only collected at a frequency of 6.6%.
- Phase IIB Groundwater - The SAP specifies a collection frequency of 10%; however, field duplicates were only collected at a frequency of 3.6%.

For OU3 samples collected in the 10 investigation phases from 2007 through 2012 (as shown in **Tables 1-3**), 48 field duplicate pairs were collected for samples prepared and analyzed by PLM, and 84 field duplicate pairs were collected for samples prepared and analyzed by TEM.

3.1.5.1 TEM

For the TEM field duplicates, the LA concentration estimates derived from the original and duplicate samples were compared using the method for comparison of two Poisson rates described by Nelson (1982), based on a 90% confidence interval (CI).

Of the 84 duplicate pairs collected for TEM analysis, 32 of the original-duplicate pairs (38%) summarized in **Table 5** below were statistically different from each other, suggesting that reproducibility of water, tree bark, and duff results (even within a small sampling scale) is difficult due to the inherent sampling variability within the medium.

Table 5 - TEM Field Duplicates Outside Reference Criteria

				Parent Sample			Field Duplicate			
Investigation	Media	Parent ID	Field Dup. ID	Struct. Count	Analytical Sensitivity [a]	Conc [a]	Struct. Count	Analytical Sensitivity [a]	Conc [a]	Poisson Ratio Rate Comparison (CI=90%)
Phase I	Duff	P1-00103	P1-00104	7	9.18E+06	6.43E+07	0	9.14E+06	<LOD	0.00E+00
		P1-00122	P1-00126	8	6.28E+06	5.02E+07	0	9.00E+06	<LOD	1.18E+08
		P1-00177	P1-00178	4	9.11E+06	3.64E+07	61	4.24E+06	2.59E+08	1.32E+05
		P1-00115	P1-00116	25	8.98E+06	2.25E+08	14	8.41E+06	1.18E+08	5.66E+05
	Tree Bark	P1-00113	P1-00114	51	1.53E+04	7.80E+05	21	8.73E+03	1.83E+05	1.03E+05
		P1-00121	P1-00125	53	3.06E+04	1.62E+06	14	9.40E+03	1.32E+05	6.77E+07
		P1-00175	P1-00176	51	9.79E+03	4.99E+05	53	1.99E+04	1.05E+06	3.49E+05
		P1-00071	P1-00072	8	5.09E+03	4.07E+04	51	1.11E+04	5.66E+05	8.96E+05
		P1-00075	P1-00076	52	8.15E+04	4.24E+06	6	8.99E+03	5.39E+04	2.14E+08
		P1-00153	P1-00154	23	9.40E+03	2.16E+05	11	9.40E+03	1.03E+05	3.59E+06
Phase IIA	Surface Water	P2-00012	P2-00013	31	9.96E+05	3.09E+07	26	1.99E+05	5.17E+06	3.32E+07
		P2-00091	P2-00093	28	9.96E+05	2.79E+07	34	1.99E+06	6.77E+07	9.42E+06
		P2-00313	P2-00314	26	6.43E+04	1.67E+06	6	7.97E+04	4.78E+05	3.73E+06
		P2-00346	P2-00347	0	4.98E+04	<LOD	7	4.98E+04	3.49E+05	2.82E+07
		P2-00351	P2-00352	27	1.05E+05	2.84E+06	25	5.53E+04	1.38E+06	6.18E+07
		P2-00802	P2-00803	25	7.66E+04	1.92E+06	18	4.98E+04	8.96E+05	1.88E+08
		P2-00930	P2-00931	7	4.98E+04	3.49E+05	22	4.98E+04	1.10E+06	0.00E+00
Phase IVB	Surface Water	P4-50043	P4-50046	119	9.96E+05	1.19E+08	215	9.96E+05	2.14E+08	1.18E+08
		P4-50097	P4-50100	100	5.69E+05	5.69E+07	107	3.32E+05	3.55E+07	1.32E+05
		P4-50124	P4-50127	101	5.86E+04	5.92E+06	45	7.97E+04	3.59E+06	5.66E+05
		P4-50151	P4-50154	100	4.43E+05	4.43E+07	100	1.42E+05	1.42E+07	1.03E+05
		P4-50181	P4-50184	0	7.97E+04	<LOD	100	3.32E+05	3.32E+07	6.77E+07
		P4-50208	P4-50211	100	1.11E+05	1.11E+07	104	2.49E+05	2.59E+07	3.49E+05
		P4-50235	P4-50238	100	4.98E+05	4.98E+07	104	9.06E+04	9.42E+06	8.96E+05
Phase VA	Surface Water	P5-10067	P5-10068	25	5.33E+04	1.33E+06	1	3.57E+04	3.57E+04	2.14E+08
		P5-20085	P5-20087	5	4.62E+04	2.31E+05	27	1.38E+05	3.73E+06	3.59E+06
Phase VB	Pore Water	P5-20007	P5-20008	39	6.92E+07	2.70E+09	57	6.92E+07	3.94E+09	3.32E+07
		P5-20097	P5-20102	56	1.66E+06	9.30E+07	34	8.30E+05	2.82E+07	9.42E+06
	Surface Water	P5-20018	P5-20019	0	8.49E+04	<LOD	65	1.64E+06	1.07E+08	3.73E+06
		P5-20225	P5-20226	25	9.96E+05	2.49E+07	62	9.96E+05	6.18E+07	2.82E+07
		P5-20261	P5-20262	26	1.15E+05	2.99E+06	25	3.08E+05	7.70E+06	6.18E+07
Commercial Logging	Duff	CL-3-0003	CL-3-0005	66	9.26E+06	6.11E+08	32	5.88E+06	1.88E+08	1.88E+08

[a] tree bark sensitivity units: $(cm)^{-2}$
water sensitivity units: $(L)^{-1}$
duff sensitivity units: $(g)^{-1}$

At least three of the duplicate pairs listed in **Table 5** (P4-50181/ P4-50184, P5-20018/ P5-20019, and P5-10067/P5-10068), appear to be outside of acceptance criteria due to possible mislabeling in the field or misidentification by the laboratory, which is part of a larger investigation described in a technical memorandum to EPA on August 30, 2013 (**see Attachment 1**).

3.1.5.2 PLM

3.1.5.2.1 PLM-VE

A field duplicate for soil is an independent sample of soil collected at the same location or coordinates and at the same time as the primary sample. Field duplicate results analyzed by PLM-VE are ranked as concordant (in agreement) if both the original sample result and the field duplicate result report the same semi-quantitative classification. Results are ranked as weakly discordant if the original sample result and the field duplicate result differ by one semi-quantitative classification (e.g., Bin A vs. Bin B1). Results are ranked as strongly discordant if the original sample result and the field duplicate result differ by more than one semi-quantitative classification (e.g., Bin A vs. Bin B2).

Table 6 summarizes the results of the original and field PLM-VE duplicate samples for forest soils, sediments, and mine waste collected from five investigative phases between 2007 and 2012. Twenty-five (25) of the 31 original duplicate pairs were found to be in concordance (80.6%). Six field duplicates were ranked as discordant, however the results were only weakly discordant. This may be due to analytical variability, but might also arise from authentic heterogeneity between the samples.

Table 6 - PLM-VE Field Duplicate Concordance Summary

			Laboratory Duplicate Results			
			Bin A (ND)	Bin B1 (Tr)	Bin B2 (<1%)	Bin C (≥1%)
			A	B1	B2	C
Original Sample Results	Bin A (ND)	A	9	1	0	0
	Bin B1 (Tr)	B1	0	7	2	0
	Bin B2 (<1%)	B2	0	2	10	0
	Bin C (≥1%)	C	0	0	1	5
Total Pairs			31			
N Concordant			25			
N Weakly Discordant			6			
N Strongly Discordant			0			
Concordant			(81%)			
Weakly Discordant			(19%)			
Strongly Discordant			(0%)			

3.1.5.2.2 PLM-GRAV

Of the 16 duplicate pairs collected for PLM-GRAV analysis, six pairs were reported as non-detect (ND), five pairs were reported as having trace (tr) levels of LA, three pairs were report as trace and >1% LA, and two pairs were reported as trace and ND. Because asbestos present in coarse samples is typically scrapped from material that cannot be passed through a ¼ inch

sieve, which can vary between the field and field duplicate pairs, these numbers should be considered acceptable.

3.2 Preparation Quality Control

Soil samples delivered to the Troy Sample Preparation Facility (SPF) and the former CDM Close Support Facility (CSF) are processed in accordance with latest revision of SOP ISSI-LIBBY-01, which includes processes for drying, splitting, sieving, grinding, and archiving soils. Once processed, the resulting fine ground and/or coarse fractions are submitted for analysis by the Libby-specific PLM methods (PLM-GRAV and PLM-VE). The purpose of grinding the samples to a uniform size prior to shipping for analysis is to remove the variability of having each of the laboratories grind their own sample. In order to ensure proper sample handling and decontamination of soil/sediment sample preparation equipment at the former CDM CSF and Troy SPF, Preparation QC samples are also collected. These samples are assigned unique field identifiers and are submitted blindly to the analytical laboratories along with the field samples. Two types of preparation QC samples were utilized for PLM analyses at the preparation facilities: preparation blanks (i.e., drying and grinding) and preparation duplicates. Of the 437 soil/sediment samples collected at OU3 for PLM-VE analysis from 2007 through 2012 for phases I, II, and V, 82 (18.8%) preparation QC duplicate and blank samples were collected (**see Table 7**).

Table 7 - PLM-VE QC

				Prep Duplicates		Grinding Blanks		Drying Blanks	
Facility	**Investigation**	**Media**	**Field Samples**	**No. of Samples**	**%**	**No. of Samples**	**%**	**No. of Samples**	**%**
CSF	Phase I	Soil	174	14	8.0%	9	5.2%	0	0.0%
	Phase IIA	Soil	176	17	9.7%	18	10.2%	14	8.0%
	Phase IIC	Soil	16	1	6.3%	1	6.3%	1	6.3%
SPF	Phase VA	Sediment	6	0	0.0%	0	0.0%	0	0.0%
	Phase VB	Sediment	65	2	3.1%	2	3.1%	3	4.6%
		Totals	**437**	**34**	**7.8%**	**30**	**6.9%**	**18**	**4.1%**

Of the 194 soil/sediment samples collected at OU3 for PLM-GRAV analysis for phases I, II, and V, 16 (8.2%) preparation QC duplicate samples were collected (**see Table 8**).

Table 8 - PLM-GRAV QC

				Prep Duplicate	
Facility	**Investigation**	**Media**	**Field Samples**	**No. of Samples**	**%**
CSF	Phase I	Solid	146	12	8.2%
	Phase IIA	Solid	27	3	11.1%
	Phase IIC	Solid	9	1	11.1%
SPF	Phase VA	Sediment	0	0	NA
	Phase VB	Sediment	12	0	0.0%
		Totals	**194**	**16**	**8.2%**

3.2.1 Preparation Blanks (Drying Blanks and Grinding Blanks)

3.2.1.1 Drying Blanks

Drying blanks consist of aliquots of asbestos-free quartz sand processed with each batch of field samples (i.e., group of routine and QC samples that are prepared for analysis at the same time). Drying blanks are used to determine if cross-contamination is occurring during sample processing (i.e., drying sieving, grinding, and splitting). A total of 18 drying blanks were analyzed from 2007 through 2012, with all reported as non-detect (Bin A) by PLM-VE. These results suggest that the procedures utilized within the preparation laboratory did not introduce LA contamination.

3.2.1.2 Grinding Blanks

Grinding blanks consist of asbestos-free quartz sand processed at a frequency of one per day. Like the drying blanks, grinding blanks are used to determine if cross-contamination has occurred during or after the grinding process. A total of 30 grinding blanks were analyzed from 2007 through 2012, with all reported as non-detect (Bin A) by PLM-VE.

Note that the number of preparation blanks recorded in **Table 7** does not reflect all of the preparation blanks prepared at the preparation facilities, but only those prepared on the days that OU3 samples were processed. Additional preparation blanks were processed with samples from other Libby OUs.

3.2.2 Preparation Duplicates

Preparation duplicates are created by dividing a sample into two parts after drying but prior to sieving and grinding, and are prepared at a frequency of 5%. Comparison of the preparation duplicate results with the paired original field sample results helps to evaluate the variability that that may occur during preparation and analysis.

3.2.2.1 PLM-VE

Similar to field duplicates, preparation duplicates for PLM-VE are ranked as concordant if both the original sample results and the preparation duplicate results display the same semi-quantitative PLM-VE classification. As shown in **Tables 9** and **10** below, a total of 30 preparation duplicates prepared and analyzed by PLM-VE; 28 at the CSF and 2 at the SPF. Of the 28 preparation duplicates at the CSF, 21 (75%) were concordant and seven (25%) were weakly discordant, and at the SPF both of the preparation duplicates (100%) were concordant. These results suggest that the PLM-VE results are generally reproducible and reliable and are not greatly influenced by differences in laboratory preparation and analysis techniques.

Table 9 - CSF Preparation Duplicate Summary

			Lab Duplicate Results			
			Bin A (ND)	Bin B1 (Tr)	Bin B2 (<1%)	Bin C (≥1%)
			A	B1	B2	C
Original Sample Results	Bin A (ND)	A	6	1	0	0
	Bin B1 (Tr)	B1	0	9	1	0
	Bin B2 (<1%)	B2	0	3	10	1
	Bin C (≥1%)	C	0	0	1	3

Table 9 - CSF Preparation Duplicate Summary

		Lab Duplicate Results			
		Bin A (ND)	Bin B1 (Tr)	Bin B2 (<1%)	Bin C (≥1%)
Total Pairs		28			
N Concordant		21			
N Weakly Discordant		7			
N Strongly Discordant		0			
Concordant		(75%)			
Weakly Discordant		(25%)			
Strongly Discordant		(0%)			

Table 10 - SPF Preparation Duplicate Summary

			Lab Duplicate Results			
			Bin A (ND)	Bin B1 (Tr)	Bin B2 (<1%)	Bin C (≥1%)
			A	B1	B2	C
Original Sample Results	Bin A (ND)	A	0	0	0	0
	Bin B1 (Tr)	B1	0	0	0	0
	Bin B2 (<1%)	B2	0	0	0	0
	Bin C (≥1%)	C	0	0	0	2
Total Pairs			2			
N Concordant			2			
N Weakly Discordant			0			
N Strongly Discordant			0			
Concordant			(100%)			
Weakly Discordant			(0%)			
Strongly Discordant			(0%)			

3.2.2.2 PLM-GRAV

Of the four preparation duplicate pairs collected for PLM-GRAV analysis, one pair was reported as having trace (tr) levels of LA, two pairs were report as trace and >1% LA, and for one set the a coarse fraction was not collected for the original sample. Because asbestos present in coarse samples is typically scrapped from material that cannot be passed through a ¼ inch sieve, of which there might be varying amounts between the field and field duplicate pairs, these numbers should be considered acceptable.

As noted above for the preparation blanks, the number of preparation duplicates recorded in **Table 7** does not reflect all of the preparation duplicates prepared at the preparation facilities, but only those prepared on the days that OU3 samples were processed. Additional preparation blanks were processed with samples from different Libby OUs.

3.3 Laboratory Analysis Quality Control

A variety of laboratory-based QC analyses are performed with TEM and PLM sample analyses to help ensure the quality of data. The results of laboratory QC applied to OU3 samples collected for all phases and analyzed between 2007 and 2012 are described in the sections below.

3.3.1 TEM Laboratory QC

The laboratory QC requirements for TEM analyses at the Libby OU3 site are patterned after the requirements set forth by NVLAP, and include:

- Laboratory blanks
- Recounts (i.e., recount same, recount different, and verified analyses)
- Re-preparations
- Inter-laboratory analyses

Table 11 provides a summary of the number and frequency at which laboratory QC analyses were performed on a program-wide basis by investigation phase.

Table 11 - TEM Laboratory QC Summary

Investigation	Media	Field Samples	Lab Blanks*		Re-preparations		Recount Different		Recount Same		Inter-Laboratory		Verified	
			Blanks	%	RP	%	RD	%	RS	%	IL	%	VA	%
Phase I	Air	49	2	4.1%	1	2.0%	1	2.0%	2	4.1%	0	0.0%	0	0.0%
	Duff	81	59	72.8%	3	3.7%	1	1.2%	1	1.2%	1	1.2%	1	1.2%
	Surface Water	30	1	3.3%	1	3.3%	0	0.0%	1	3.3%	0	0.0%	0	0.0%
	Tree Bark	82	4	4.9%	3	3.7%	3	3.7%	1	1.2%	0	0.0%	0	0.0%
	Phase I Total	242	66	27.3%	8	3.3%	5	2.1%	5	2.1%	1	0.4%	1	0.4%
Phase IIA	Surface Water	290	11	3.8%	5	1.7%	9	3.1%	4	1.4%	2	0.7%	1	**0.3%**
Phase IIB	Air	82	4	4.9%	2	2.4%	3	3.7%	1	1.2%	1	1.2%	2	2.4%
	Groundwater	28	0	0.0%	0	**0.0%**	1	3.6%	0	0.0%	0	0.0%	1	3.6%
	Phase IIB Total	110	4	**3.6%**	2	1.8%	4	3.6%	1	0.9%	1	0.9%	3	2.7%
Phase IIC	Surface Water	4	0	**0.0%**	0	**0.0%**	0	**0.0%**	0	**0.0%**	0	**0.0%**	0	**0.0%**
Phase III	Air (ABS)	241	11	4.6%	6	**2.5%**	3	**1.2%**	-	---	3	1.2%	0	**0.0%**
Phase IVA	Air (ABS)	262	11	4.2%	7	2.7%	6	2.3%	-	---	8	3.1%	0	0.0%
	Tree Bark	5	0	0.0%	1	20.0%	0	0.0%	-	---	1	20.0%	0	0.0%
	Phase IVA Total	267	11	4.1%	8	**3.0%**	6	2.2%	-	---	9	3.4%	0	**0.0%**
Phase IVB	Surface Water	104	1	1.0%	0	**0.0%**	0	**0.0%**	1	1.0%	2	1.9%	0	**0.0%**
Phase VA	Air (ABS)	3	0	0.0%	0	0.0%	0	0.0%	0	0.0%	0	0.0%	0	0.0%
	Surface Water	88	1	1.1%	1	1.1%	0	0.0%	0	0.0%	0	0.0%	1	1.1%
	Phase VA Total	91	1	1.1%	1	**1.1%**	0	**0.0%**	0	**0.0%**	0	**0.0%**	1	1.1%
Phase VB	Pore Water	49	0	0.0%	3	6.1%	1	2.0%	0	0.0%	2	4.1%	0	0.0%
	Surface Water	124	4	3.2%	1	0.8%	2	1.6%	0	0.0%	1	0.8%	1	0.8%
	Phase VB Total	173	4	2.3%	4	**2.3%**	3	**1.7%**	0	**0.0%**	3	**1.7%**	1	**0.6%**
CL	Air (ABS)	17	0	0.0%	0	0.0%	0	0.0%	0	0.0%	1	5.9%	0	0.0%
	Duff	6	3	50.0%	1	16.7%	0	0.0%	0	0.0%	1	16.7%	0	0.0%
	Tree Bark	6	3	50.0%	0	0.0%	0	0.0%	0	0.0%	1	16.7%	0	0.0%
	Phase CL Total	29	6	20.7%	1	**3.4%**	0	**0.0%**	0	**0.0%**	3	10.3%	0	**0.0%**

* For duff samples the blank total includes drying and filtration blanks that were prepared as part of initial method development

In **Table 11** above, the TEM laboratory QC sample frequency goals, as specified in the investigative SAPs, were not met for those percentages highlighted in bold. Note that the frequency goals provided in the SAPs are not media-specific, and therefore the frequency criteria are applied to the totals for each of the investigations. For the year 2012 (Phase V), the

primary laboratory used by EPA for TEM analysis from OU3 stopped assigning project-specific QC analyses for OU3 samples at the required frequency, which explains the absence of QC for most media in 2012. A more detailed explanation of this discrepancy is described in the technical memorandum submitted to EPA on August 30, 2013 (**see Attachment 1**).

3.3.1.1 Laboratory Blanks

Laboratory blanks are prepared from new, unused filters and analyzed using the same procedures used to analyze the associated field samples. The purpose of a laboratory blank is to determine the presence of any significant sources of asbestos contamination during sample preparation and analysis in the TEM laboratory. As specified in Libby Laboratory Modification LB-000029, laboratory blanks are to be analyzed at a frequency of 4%; however, as shown in **Table 11**, not every sampling program achieved this goal. Although the overall frequency across programs is greater than 4%, this number is biased high due to the inclusion of the drying and filtration blanks prepared with duff samples. When the extra blanks prepared with duff samples are excluded, the total number of preparation blanks is 46, for a program-wide frequency of 3.4%.

Including filtration and drying blanks, a total of 115 TEM laboratory blanks were analyzed across all sampling programs. No asbestos structures were observed in any of the laboratory blank samples prepared and analyzed. These results suggest that the sample preparation and analysis procedures performed by the laboratories did not introduce asbestos contamination.

3.3.1.2 Recounts

A recount analysis is a re-examination of the original TEM grid openings to verify the reported asbestos structure counts and characteristics. The following types of recount analyses were performed by the analytical laboratories during TEM determinations:

- Recount Same (RS) – This is a TEM analysis where the original grid openings are re-examined by the same microscopist who performed the initial examination.
- Recount Different (RD) – This is a TEM analysis where the original grid openings are re-examined by a different microscopist within the same laboratory who did not perform the initial examination.
- Verified Analysis (VA) – This analysis is similar to an RD but has different documentation requirements. A VA must be recorded in accordance with the 1994 protocol.

Recount analyses were compared with the original analysis on a grid opening-by-grid opening and structure-by-structure basis. Only those grid openings that were able to be re-examined during the recount analysis were included in this evaluation. The degree of concordance between the original analysis and the recount analysis was evaluated based on the total number of countable LA structures observed for each grid opening that was re-examined. The concordance criteria, which are summarized below, are specified in the most recent revision of Libby Laboratory Modification LB-000029:

- Number of LA structures within each grid opening - For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10 percent (%).

- Asbestos class of structure (LA, OA, or CH) - The class of structure must agree 100% on CH vs. amphibole. For assignment of amphiboles to LA or OA bins, there must be agreement on at least 90% of all amphibole structures.
- Structure Length - Fibers and bundles must agree within 0.5 microns (µm) or 10%, whichever is less stringent. Clusters and matrices must agree within 1 µm or 20%, whichever is less stringent.
- Structure Width - Fibers and bundles must agree within 0.5 µm or 20%, whichever is less stringent. For clusters and matrices, there is no quantitative rule for concordance.

As summarized in **Table 11,** a total of 11 RS, 30 RD, 7 VA, and 24 IL analyses have been performed across all of the sampling programs, for an overall frequency of recount analyses of approximately 4.6% (0.7% RS; 1.9% RD; 0.5% VA and 1.5% IL).

Tables 12A-12E show the recount analysis results for various matrices in investigation Phases I-V by mineral class, structure length, structure width, and matched structures per grid opening.

Table 12A – Phase I Recounts

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
Water	Mineral Class	10	10	100%	Tree Bark	Mineral Class	106	106	100%
	Structure Length	10	10	100%		Structure Length	106	104	98%
	Structure Width	10	10	100%		Structure Width	106	99	93%
	Structure per GO	8	8	100%		Structure per GO	16	12	75%
Duff	Mineral Class	118	118	100%	**Total**	**Mineral Class**	**234**	**234**	**100%**
	Structure Length	118	111	94%		**Structure Length**	**234**	**225**	**96%**
	Structure Width	118	117	99%		**Structure Width**	**234**	**226**	**97%**
	Structure per GO	28	24	86%		**Structure per GO**	**52**	**44**	**85%**
Air	Mineral Class	--	--	--					
	Structure Length	--	--	--					
	Structure Width	--	--	--					
	Structure per GO	--	--	--					

Note: For the air sample summarized above no structures were detected in either the original or recount analysis.

With the exception of the structures per grid opening for Tree Bark, all of the above results fall into either the good or acceptable ranges. Of the 16 Grid Openings (GOs) analyzed for Tree Bark, only 12 (75%) met the specified criteria of ≥ 85%, which is considered poor.

Table 12B – Phase II Recounts

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
Air	Mineral Class	22	22	100%	Water	Mineral Class	327	327	100%
	Structure Length	22	21	95%		Structure Length	327	287	88%
	Structure Width	22	22	100%		Structure Width	327	324	99%
	Structure per GO	13	10	77%		Structure per GO	**110**	**99**	**90%**
					Total	**Mineral Class**	**349**	**349**	**100%**
						Structure Length	**349**	**308**	**88%**
						Structure Width	**349**	**346**	**99%**

Table 12B – Phase II Recounts

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
						Structure per GO	**123**	**109**	**89%**

With the exception of the structures per grid opening for air, all of the above results fall into either the good or acceptable ranges. Of the 13 Grid Openings (GOs) analyzed for air, only 10 (77%) met the specified criteria of ≥ 85%, which is considered poor.

Table 12C – Phase III Recounts

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
Air	Mineral Class	3	3	100%	**Total**	**Mineral Class**	**3**	**3**	**100%**
	Structure Length	3	3	100%		**Structure Length**	**3**	**3**	**100%**
	Structure Width	3	3	100%		**Structure Width**	**3**	**3**	**100%**
	Structure per GO	3	3	100%		**Structure per GO**	**3**	**3**	**100%**

Table 12D – Phase IV Recounts

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
Air	Mineral Class	54	54	100%	Water	Mineral Class	108	108	100%
	Structure Length	54	51	94%		Structure Length	108	95	88%
	Structure Width	54	54	100%		Structure Width	108	104	96%
	Structure per GO	48	46	96%		Structure per GO	10	8	80%
					Total	**Mineral Class**	**162**	**162**	**100%**
						Structure Length	**162**	**146**	**90%**
						Structure Width	**162**	**158**	**98%**
						Structure per GO	**58**	**54**	**93%**

Table 12E – Phase V Recounts

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
Water	Mineral Class	109	109	100%	**Total**	**Mineral Class**	**109**	**109**	**100%**
	Structure Length	109	109	100%		**Structure Length**	**109**	**109**	**100%**
	Structure Width	109	108	99%		**Structure Width**	**109**	**108**	**99%**
	Structure per GO	50	49	98%		**Structure per GO**	**50**	**49**	**98%**

3.3.1.3 Re-preparations

A re-preparation is a TEM analysis where new grids are prepared using a new portion of the same field sample filter used to prepare the original grids. The results are compared to those from the original analysis based on the Poisson rate ratio method recommended by Nelson (1982). Re-preparations provide information on analysis precision, as well as within-filter variability. Re-preparations were prepared for 35 samples across each sampling program for water, air, tree bark, and duff, with the frequency of each summarized in **Table 11** above. The overall frequency of re-preparation analyses was 2.3%, which is above the minimum frequency requirement of 1.0%.

Table 13 summarizes the results of those re-preparation analyses that were statistically different from the original analysis for water and tree bark samples. The five samples listed in the Table represent 14% of the 35 re-preparations performed across all media types, indicating that 86% of the re-preparations performed were within the established criteria. When compared to the program-wide goals for Good (>95%), Acceptable (90-95%), or Poor (<90%), the 86% would indicate "Poor", which should prompt investigation and possible corrective action.

Table 13 - Statistical Comparison of TEM Re-preparation Analyses

Investigation	Media	Media	First Evaluation (Rate 1)			Re-prep. Evaluation (Rate 2)			Poisson Ratio Rate Comparison (CI=90%)
			Struct. Count	Sensitivity [a]	Conc [a]	Struct. Count	Sensitivity [a]	Conc [a]	
Phase IIA	Surface Water	P2-00208	0	4.98E+04	<LOD	35	1.99E+06	6.97E+07	[0-0] Rate 1<Rate 2
	Surface Water	P2-00804	17	4.98E+04	8.47E+05	83	4.98E+04	4.13E+06	[0.13-0.32] Rate 1<Rate 2
Phase IVA	Pore Water	P5-20001	42	6.92E+07	2.91E+09	27	5.54E+07	1.50E+09	[1.26-3.02] Rate 1>Rate 2
Phase VB	Pore Water	P5-20054	43	1.66E+06	7.14E+07	41	4.62E+05	1.89E+07	[2.57-5.54] Rate 1>Rate 2
	Tree Bark	SP4-00787	56	5.16E+04	2.89E+06	54	1.61E+04	8.69E+05	[2.38-4.63] Rate 1>Rate 2

[a] tree bark sensitivity units: $(cm)^{-2}$
water sensitivity units: $(L)^{-1}$

3.3.1.4 TEM Inter-laboratory Analyses

Samples for TEM inter-laboratory analyses were selected in accordance with the most recent revision of Laboratory Modification LB-000029. Once selected, the list was provided to each of the participating laboratories, who then retrieved the sample(s) from their archive storage, prepared the necessary TEM grids, analyzed the samples, prepared the necessary paperwork, and shipped the grids to the laboratory selected to perform the inter-laboratory analyses. The criteria for inter-laboratory analyses are the same as those for the other recount analyses, which are described in **Section 3.3.1.2** above. The following tables (**Tables 14A-14E**) provide a summary of the results by both investigation phase and media:

Table 14A - Phase II Inter-laboratory Analyses

Results for Matched LA Structures									
Matrix	Attribute	Total	Pass	%	Matrix	Attribute	Total	Pass	%
Air	Mineral Class	13	13	100%	Water	Mineral Class	86	85	99%
	Structure Length	13	6	46%		Structure Length	86	32	37%
	Structure Width	13	6	46%		Structure Width	86	84	98%
	Structure per GO	4	3	75%		Structure per GO	15	4	27%
					Total	**Mineral Class**	**99**	**98**	**99%**
						Structure Length	**99**	**38**	**38%**
						Structure Width	**99**	**90**	**91%**
						Structure per GO	**19**	**7**	**37%**

With the exception of the following, all of the above results fall into either the good or acceptable ranges:

- Air - The percentage of the structure length and widths passing the specified criteria is not ≥80%, which is considered poor.
- Air - The percentage of GOs with structure counts passing the specified criteria is not ≥85%, which is considered poor.

- Water - The percentage of the structure lengths passing the specified criteria is not ≥80%, which is considered poor.

Table 14B - Phase III Inter-laboratory Analyses

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
Air	Mineral Class	2	2	100%	**Total**	**Mineral Class**	**2**	**2**	**100%**
	Structure Length	2	2	100%		**Structure Length**	**2**	**2**	**100%**
	Structure Width	2	2	100%		**Structure Width**	**2**	**2**	**100%**
	Structure per GO	2	2	100%		**Structure per GO**	**2**	**2**	**100%**

Table 14C - Phase IV Inter-laboratory Analyses

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
Air	Mineral Class	41	38	93%	Tree Bark	Mineral Class	65	62	95%
	Structure Length	41	35	85%		Structure Length	65	54	83%
	Structure Width	41	39	95%		Structure Width	65	60	92%
	Structure per GO	45	29	64%		Structure per GO	4	0	0%
Water	Mineral Class	114	114	100%	**Total**	**Mineral Class**	**220**	**214**	97%
	Structure Length	114	100	88%		**Structure Length**	**220**	**189**	86%
	Structure Width	114	112	98%		**Structure Width**	**220**	**211**	96%
	Structure per GO	4	3	75%		**Structure per GO**	**53**	**32**	**60%**

With the exception of the following, all of the above results fall into either the good or acceptable ranges:

- Air (ABS) - The percentage of GOs with structure counts passing the specified criteria is not ≥85%, which is considered poor.
- Water - The percentage of GOs with structure counts passing the specified criteria is not ≥85%, which is considered poor.
- Tree Bark - The percentage of GOs with structure counts passing the specified criteria is not ≥85%, which is considered poor.

It should be noted that the results from the inter-laboratory analysis of tree bark sample SP4-00795 are not included in this summary. As explained in the report narrative, it was determined that the laboratory that performed the original analysis (RP) used standard ISO 10312 counting rules, which do not take into account the best estimate of visible length and width described in Laboratory Modification LB-000016H. As a result of this error, the analyses could not be compared.

Table 14D - Phase V Inter-laboratory Analyses

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
Water	Mineral Class	111	105	95%	**Total**	**Mineral Class**	**111**	**105**	**95%**
	Structure Length	111	98	88%		**Structure Length**	**111**	**98**	**88%**
	Structure Width	111	111	100%		**Structure Width**	**111**	**111**	**100%**
	Structure per GO	10	3	30%		**Structure per GO**	**10**	**3**	**30%**

With the exception of the structures per grid opening, all of the above results fall into either the good or acceptable ranges. Of the 10 GOs analyzed, only 3 (30%) met the specified criteria of ≥ 85%, which is considered poor.

Table 14E - Commercial Logging (CL) Inter-laboratory Analyses

Results for Matched LA Structures									
Matrix	**Attribute**	**Total**	**Pass**	**%**	**Matrix**	**Attribute**	**Total**	**Pass**	**%**
Air	Mineral Class	19	16	84%	Tree Bark	Mineral Class	38	38	100%
	Structure Length	19	18	95%		Structure Length	38	34	89%
	Structure Width	19	18	95%		Structure Width	38	38	100%
	Structure per GO	10	6	60%		Structure per GO	4	3	75%
Duff	Mineral Class	79	76	96%	**Total**	**Mineral Class**	**130**	**136**	**96%**
	Structure Length	79	63	80%		**Structure Length**	**115**	**136**	**85%**
	Structure Width	79	78	99%		**Structure Width**	**134**	**136**	**99%**
	Structure per GO	4	1	25%		**Structure per GO**	**18**	**10**	**56%**

With the exception of the following, all of the above results fall into either the good or acceptable ranges:

- Air (ABS) - The percentage of GOs with structure counts passing the specified criteria is not ≥85%, which is considered poor.
- Duff - The percentage of GOs with structure counts passing the specified criteria is not ≥85%, which is considered poor.
- Tree Bark - The percentage of GOs with structure counts passing the specified criteria is not ≥85%, which is considered poor.

Table 15 below provides an additional, investigation-specific summary of instances of structures identified in the original analyses, but not in the recount analyses and vice versa.

Table 15 - Inter-laboratory Structure Discrepancies

Investigation	Media	Not in Original (RP)	Not in Interlab (IL)
Phase II	Air	0	3
	Water	13	19
Phase IV	Air	31	2
	Tree Bark	14	1
Phase IV	Water	29	5
Phase VB	Water	21	0
CL	Duff	27	4
	Tree Bark	5	13

Of the 11 inter-laboratory analyses performed by the laboratories from 2010 through 2012, seven samples (64%) required reconciliation due to discordant results between the original (RP) and inter-laboratory (IL) analyses. The reasons for discordance in the results provided by the laboratories include:

- The use by laboratories of standard ISO 10312 counting rules and not the modified rules described in the most recent revision of Laboratory Modification LB-000016.
- Failure to record “close call” non-asbestos material (NAM) structures, which could be identified as LA by the laboratory performing the inter-laboratory analysis.

- Measurement inaccuracies, most of which were related to the lengths of structures intersecting countable grid bars.
- The presence of damaged grid openings that could not be re-analyzed, resulting in incomplete inter-laboratory analyses.
- The interpretation of EDS spectrum as it applies to the identification of LA versus a "close call" NAM due to the presence of slightly higher aluminum (Al) and relatively lower calcium (Ca) peaks.

In addition to the above, a higher than normal incidence of damaged grid openings were observed during the 2010 and 2011 inter-lab studies. It was determined that this problem was isolated to those laboratories using grids with a larger than normal grid openings (i.e., 0.0130mm^2 versus 0.006mm^2). In response to this observation, corrective action was applied which included a requirement to use grids with smaller grid openings. This resulted in a dramatic decrease in the frequency of damaged grid openings in the 2012 study. A detailed explanation of this observation and eventual corrective action is provided in the technical memorandum submitted to EPA on August 30, 2013. Although this issue does not directly affect field sample analyses, it could affect associated QC analyses and the ability to reanalyze samples at a later date if needed (**see Attachment 1**).

3.3.2 PLM Laboratory QC

Three types of laboratory-based QC analyses are performed for OU3 samples analyzed by PLM-VE: laboratory duplicates, inter-laboratory analyses, and the analyses of PESs.

3.3.2.1 Laboratory Duplicates

A laboratory duplicate is a reanalysis of a sample within the same laboratory. There are two types of laboratory duplicates performed for PLM-VE:

- Laboratory Duplicate Self-check (LDS) – A reanalysis of a client sample by the same analyst.
- Laboratory Duplicate Cross-check (LDC) – A reanalysis of a client sample by a different analyst within the same laboratory.

Table 16 provides a summary of the frequency at which these analyses were performed by phase, and program-wide. As a whole, the laboratories exceeded the frequency goal for LDS and IL analyses, but fell short of the frequency goal for LDCs, as indicated in **Table 16**. It should also be noted that these analyses were not always performed at the specified frequencies by the individual laboratories.

Table 16 – PLM-VE Lab QC

			Lab Dup-Cross Check (LDC) (Freq. Goal 8%)		Lab Dup Self Check (LDS) (Freq. Goal 2%)		Inter-laboratory (IL) (Freq. Goal 1.0%)	
Investigation	**Media**	**Field Samples**	**LDC**	**%**	**LDS**	**%**	**IL**	**%**
Phase I	Forest Soil	82	--	--	21	25.6%	--	--
	Mine waste	42	--	--	0	0.0%	--	--
	Sediment	27	--	---	0	0.0%	--	--
	Soil	23	--	---	1	4.3%	--	--

Table 16 – PLM-VE Lab QC

			Lab Dup-Cross Check (LDC) (Freq. Goal 8%)		Lab Dup Self Check (LDS) (Freq. Goal 2%)		Inter-laboratory (IL) (Freq. Goal 1.0%)	
Investigation	**Media**	**Field Samples**	**LDC**	**%**	**LDS**	**%**	**IL**	**%**
Phase IIA	Sediment	128	17	13.3%	0	0.0%	15	11.7%
	Soil	48	5	10.4%	0	0.0%	0	0.0%
Phase IIC	Sediment	13	2	15.4%	0	0.0%	1	7.7%
	Soil	3	1	33.3%	0	0.0%	0	0.0%
Phase VA	Sediment	6	1	16.7%	0	0.0%	2	33.3%
Phase VB	Sediment	65	1	1.5%	3	4.6%	1	1.5%
		437	**27**	**6.2%**	**25**	**5.7%**	**19**	**4.3%**

A total of 70 laboratory duplicate analysis pairs, made up of 27 LDC, 25 LDS, and 19 IL analyses, were performed from 2007 through 2012. As illustrated in **Table 17** below, 100% of the LDS results were concordant with the original results.

Table 17 - PLM-VE LDS Summary

			Lab Duplicate Results			
			Bin A (ND)	Bin B1 (Tr)	Bin B2 (<1%)	Bin C (≥1%)
			A	B1	B2	C
Original Sample Results	Bin A (ND)	A	22	1	0	0
	Bin B1 (Tr)	B1	0	0	0	0
	Bin B2 (<1%)	B2	0	0	0	0
	Bin C (≥1%)	C	0	0	0	3
Total Pairs			25			
N Concordant			25			
N Weakly Discordant			0			
N Strongly Discordant			0			
Concordant			(100%)			
Weakly Discordant			(0%)			
Strongly Discordant			(0%)			

As shown in **Table 18** below, for the LDC results, all but one of the results were concordant with the original results. The one discordant PLM-VE LDC result reported was only weakly discordant.

Table 18 - PLM-VE LDC Summary

			Lab Duplicate Results			
			Bin A (ND)	Bin B1 (Tr)	Bin B2 (<1%)	Bin C (≥1%)
			A	B1	B2	C
Original Sample Results	Bin A (ND)	A	6	1	0	0
	Bin B1 (Tr)	B1	0	6	0	0
	Bin B2 (<1%)	B2	0	0	8	0
	Bin C (≥1%)	C	0	0	0	7

Table 18 - PLM-VE LDC Summary

		Lab Duplicate Results			
		Bin A (ND)	Bin B1 (Tr)	Bin B2 (<1%)	Bin C (≥1%)
		A	B1	B2	C
Total Pairs		27			
N Concordant		26			
N Weakly Discordant		1			
N Strongly Discordant		0			
Concordant		(96%)			
Weakly Discordant		(4%)			
Strongly Discordant		(0%)			

3.3.2.2 PLM Inter-laboratory Analyses

Inter-laboratory samples are samples previously analyzed by one laboratory, which are selected by QATS for analysis by another laboratory. For OU3 PLM analyses, the IL samples were selected in accordance with the most recent revision of laboratory modification LB-000073. Once the IL samples were selected, a finely ground (FG) aliquot, which had not been analyzed, was retrieved from the sample archive at the Troy SPF and shipped to the laboratory performing the inter-laboratory analysis. Because these samples are shipped from the Troy SPF and not the originating laboratory the laboratories are not aware that the samples are being shipped for inter-laboratory analysis. **Table 19** provides a summary of the results from samples previously analyzed in 2012 and selected for inter-laboratory analysis of samples.

Table 19 - PLM-VE Inter-Laboratory Summary

			Inter-laboratory Results			
			Bin A (ND)	Bin B1 (Tr)	Bin B2 (<1%)	Bin C (≥1%)
			A	B1	B2	C
Original Sample Results	Bin A (ND)	A	5	0	0	0
	Bin B1 (Tr)	B1	0	4	1	1
	Bin B2 (<1%)	B2	0	3	5	2
	Bin C (≥1%)	C	0	0	2	5
Total Pairs			19			
N Concordant			10			
N Weakly Discordant			8			
N Strongly Discordant			1			
Concordant			(53%)			
Weakly Discordant			(42%)			
Strongly Discordant			(5%)			

The PLM-VE IL results were 53% concordant, 42% weakly discordant, and 5% strongly discordant. The most recent revision of laboratory modification LB-000073 indicate that weakly discordant results greater than 40% fall into the “poor” category, necessitating corrective action. It should be noted, however, that this evaluation of OU3 PLM-VE data represents a relatively small sample size. A better measurement of the overall quality of the PLM-VE data can be determined from the larger sample size collected for the site-wide study.

3.4 Performance Evaluation Sample Results

Performance Evaluation Samples (PES) are synthetic, man-made test samples that are prepared by "spiking" a known concentration of asbestos into a contaminant-free media. As illustrated in **Table 20** below, of these 40 PLM-VE PES pairs, 78% were concordant, 18% were weakly discordant, and 5% were strongly discordant.

Table 20 - PLM-VE PES Summary

			PES Results			
			Bin A (ND)	Bin B1 (Tr)	Bin B2 (<1%)	Bin C (≥1%)
			A	B1	B2	C
Original Sample Results	Bin A (ND)	A	8	0	0	0
	Bin B1 (Tr)	B1	0	4	1	0
	Bin B2 (<1%)	B2	0	0	12	3
	Bin C (≥1%)	C	0	2	3	16
Total Pairs			40			
N Concordant			31			
N Weakly Discordant			7			
N Strongly Discordant			2			
Concordant			(78%)			
Weakly Discordant			(18%)			
Strongly Discordant			(5%)			

4.0 Asbestos Data Validation

Data for asbestos in air, tree bark, mine waste, surface water, duff, sediment, forest soil, groundwater, and pore water were validated by the QATS Program in accordance with the applicable method, SAP Analytical Requirements Summaries, Laboratory Modifications, and QATS Libby-specific data validation SOPs, which include SOP QATS-70-094 (Validation of Polarized Light Microscopy (PLM) Data Deliverables) and SOP QATS-70-095 (Validation of Libby Transmission Electron Microscopy (TEM) Data Deliverables).

The validation process involves evaluating asbestos data based on the analytical requirements in the applicable method or SOP used by EPA for analysis of Libby OU3 samples. These include Method ISO 10312 and EPA Method 100.2 for TEM, and PLM-VE and PLM-GRAV for PLM analysis. Criteria that are evaluated and reported include Sample Receipt, Sample Preparation, Microscope Alignment, Instrument Calibrations, Stopping Rules, Structure Recording and Identification, Blank Analysis (if applicable), Recount/Re-preparation Analysis (if applicable), and Overall Assessment of Data.

Data are qualified if the daily or monthly calibrations associated with a sample set were not performed at the required frequency, or if the calibrations fail to meet method requirements. The equipment alignment and calibration documentation from each of the Libby support laboratories are provided separately on a quarterly basis. This calibration information is entered into laboratory-specific spreadsheets where the data validators can access the information and verify that the calibrations were acceptable and performed at the correct frequency.

Qualifiers for blank contamination are applied during the validation process for those blanks directly associated with field samples (i.e., provided with a particular deliverable selected for

validation). In addition to those QC analyses reviewed during the validation of select deliverables, QC analyses are also reviewed and evaluated on a program-wide basis to ensure they are both performed at the required frequency and that they are within the applicable criteria. With the exception of QC analyses directly associated with a particular set of samples, laboratory QC analyses are performed to determine the overall quality of the collective data, and not the quality of any one specific set of samples.

The data validation process also includes a comparison of the information reported on the bench sheets to the entries in the associated laboratory method-specific EDDs to ensure that the reported results are complete, compliant with the specified methodology, and accurate. These comparison discrepancies are noted in a separate table of the data validation report. A QATS Data Review Checklist is used to document the data validation process.

Selection of the five percent (5%) of sample results to validate was performed by randomly choosing sample results by laboratory, method, and media. A total of 360 field samples from 30 Laboratory Job Numbers, analyzed by five different laboratories between 2007 and 2012 were selected for validation. The Lab Jobs selected by year, laboratory, and method are listed in **Table 21** below:

Table 21 - Validated Asbestos OU3 Deliverables

Year	Laboratory	Lab Job No.	Method/Media	Number of Samples
2007	EMSL, Westmont, NJ	040726758	TEM ISO/Air	11
2007	EMSL, Westmont, NJ	040724967	TEM ISO/Tree Bark	3
	EMSL, Beltsville, MD			28
2007	EMSL, Westmont, NJ	040730897	PLM-VE/Mine Waste	5
2007	EMSL, Westmont, NJ	040730895	PLM-GRAV/Mine Waste	4
2007	EMSL, Libby, MT	270701089	TEM 100.2/Surface Water	2
2008	EMSL, Westmont, NJ	040725946	TEM ISO/Duff	20
	EMSL, Libby, MT			8
2008	EMSL, Westmont, NJ	040726269	TEM ISO/Tree Bark	25
	EMSL, Libby, MT			26
2008	EMSL, Westmont, NJ	040817340	PLM-VE/Sediment	12
2008	EMSL, Westmont, NJ	040825407	PLM-GRAV/Sediment	1
2008	EMSL, Beltsville, MD	270800192	TEM ISO/Surface Water	4
2008	EMSL, Libby, MT	270800012	PLM-GRAV/Forest Soil	44
2008	EMSL, Libby, MT	270800018	PLM-VE/Forest Soil	10
2008	EMSL, Libby, MT	270800271	TEM ISO/Surface Water	8
2008	EMSL, Libby, MT	270800714	TEM ISO/Air	10
2009	EMSL, Westmont, NJ	040725351	TEM ISO/Duff	23
2009	EMSL, Westmont, NJ	270800257	TEM ISO/Surface Water	4
	EMSL, Beltsville, MD			3
2009	Hygeia Environmental	12302090005	TEM ISO/Air	6
2009	Hygeia Environmental	12302090025	TEM ISO/Air	9
2009	Hygeia Environmental	12302080011	PLM-VE/Sediment	16
2010	EMSL, Cinnaminson, NJ	041021393	TEM ISO/Tree Bark	5
2010	EMSL, Libby, MT	271001549	TEM ISO/Water	10
2010	Hygeia Environmental	12302100026	TEM ISO/Air	15
2011	EMSL, Libby, MT	271100171	TEM ISO/Surface Water	6

Table 21 - Validated Asbestos OU3 Deliverables

Year	Laboratory	Lab Job No.	Method/Media	Number of Samples
2011	Hygeia Environmental	12302110006	TEM ISO/Surface Water	3
2012	EMSL, Cinnaminson, NJ	271200263	TEM ISO/Pore Water	3
2012	EMSL, Libby, MT	271201033	PLM-VE/Sediment	5
			PLM-GRAV/Sediment	8
2012	EMSL, Libby, MT	271201048	TEM ISO/Air	17
2012	EMSL, Libby, MT	271200347	TEM ISO/Pore Water	3
2012	EMSL, Denver, CO	271200306	TEM ISO/Pore Water	3
Total				**360**

Note that a total of 25 surface water samples collected in 2012 were included in Laboratory Job Numbers 271200263, 271200275, 271200347, and 271200306; however, validation of these samples is currently on hold pending an investigation into a possible sample mix-up during the sampling effort.

Very few OU3 asbestos data were qualified. Qualifiers were applied to only one field sample and one QC sample (recount different) of the 360 asbestos samples validated (0.56%); 99.4% of the OU3 asbestos results for samples analyzed between 2007 and 2012 required no qualification. The samples were qualified due to the failure of the laboratory to perform and/or document daily calibration activities. Several samples that did not have a daily calibration were not qualified due to the submission and review of other information suggesting acceptable instrument performance, such as spectra, daily Standard Reference Material (SRM) analysis, review of the bracketing daily alignment, evaluation of concordance between recounts or re-preparations, or the presence of structures versus non-detected results. The samples that were qualified for lack of a daily calibration are listed in **Table 22** below:

Table 22 - Qualified Samples

Laboratory	EPA Sample ID	Lab Job No.	Date Analyzed	Method/Media	Qualifier
EMSL, Westmont, NJ	P1-00115 RD	040725946	02/10/2009	TEM ISO/Duff	J*
EMSL, Denver, CO	P5-20041	271200306	07/08/2012	TEM ISO/Pore Water	J*

J* - The result (concentration) is estimated.

In addition to the 360 field samples validated, 43 blanks and 25 QC samples were validated. The QC samples are listed by type and analysis year in **Table 23** below:

Table 23 – Number of Blanks and QC Samples Validated

QC Type	Analysis Year						
Blank/QC Sample Type	**2007**	**2008**	**2009**	**2010**	**2011**	**2012**	**Total**
Laboratory Blanks	2	3	1	5		1	12
Field Blanks	2	1	2	1			6
Drying Blanks		4	17				21
Filtration Blanks		2	1	1			4
Recount Same (RS)	2	1			1		4
Recount Different (RD)	1	3					4
Repreparation (RP)	3	3	1	3			10

Table 23 – Number of Blanks and QC Samples Validated

QC Type	Analysis Year						
Blank/QC Sample Type	2007	2008	2009	2010	2011	2012	Total
Verified Analysis (VA)		1	1				2
Reconciliation (RC)		1					1
Lab Duplicate Self-check (LDS)							0
Lab Duplicate Cross-check (LDC)		2	2				4

From the data of 428 field samples, QC samples, and blanks validated, the results from one field sample and one QC sample were qualified.

The bench sheet/EDD information comparisons did reveal discrepancies due to information omissions and typographical errors, which were reported in the EDD/Bench Sheet Discrepancy Table in the Asbestos Validation Summary Reports. The discrepancies ranged from minor (i.e., typographical errors in fields that do not affect the sample results) to more severe discrepancies (i.e., typographical errors for fiber length and/or width, primary filter area, mineral identification which could affect the sample result, or date analyzed discrepancies which could affect the daily calibration verification). A total of 89 of the 360 sample results validated (24.7%) contained some type of bench sheet/EDD discrepancy. However, 86 of these 89 (97%) were minor typographical discrepancies. **Table 24** shows the number of discrepancies found in the EDD files submitted by laboratory and the analysis year:

Table 24 - Number of Discrepancies Listed in the EDD/Bench Sheet Discrepancy Table

Laboratory	2007	2008	2009	2010	2011	2012
EMSL, New Jersey	1	7	2	2	NA	4
EMSL, Beltsville, MD	12	1	0	NA	NA	NA
EMSL, Denver, CO	NA	NA	NA	NA	NA	2
EMSL, Libby, MT	2	22	0	6	3	5
Hygeia Environmental	NA	NA	4	0	1	NA

NA indicates no samples analyses from the laboratory were performed for that year.

5.0 Laboratory Audits

This section includes a summary of the results of on-site audits of laboratories and soil preparation facilities used by EPA for analytical support at the Libby OU3 site that were conducted between 2008 and 2012. During this period, a total of 14 on-site audits were performed, consisting of nine asbestos laboratory audits, three asbestos soil preparation facility (SPF) audits, and two asbestos toxicology study laboratory audits. **Table 25** lists the audits performed by laboratory/facility, audit type, and date.

Table 25 - Asbestos/Toxicology Laboratory and Soil Preparation Facility On-site Audits

Laboratory	Audit Type	Audit Date(s)
Fort Environmental Laboratories, Inc. (Stillwater, OK)	Toxicology Laboratory	11/19/2012
EMSL Analytical, Inc. (Libby, MT)	Asbestos Laboratory	08/08-09/2012
ESAT Region 8 Soil Preparation Facility (Troy, MT)	Soil Preparation Facility	08/07/2012
Hygeia Environmental, Inc. (Sierra Madre, CA)	Asbestos Laboratory	07/25-26/2012

Table 25 - Asbestos/Toxicology Laboratory and Soil Preparation Facility On-site Audits

Laboratory	Audit Type	Audit Date(s)
EMSL Analytical, Inc. (Beltsville, MD)	Asbestos Laboratory	06/28-29/2012
EMSL Analytical, Inc. (Westmont, NJ) [1]	Asbestos Laboratory	06/26-27/2012
EMSL Analytical, Inc. (Denver, CO)	Asbestos Laboratory	05/21-22/2012
Oregon State University (OSU) Aquatic Toxicology Lab.	Toxicology Laboratory	06/11/2011
CDM Close Support Facility (Denver, CO)	Soil Preparation Facility	10/02/2008
ESAT Region 8 Soil Preparation Facility (Troy, MT)	Soil Preparation Facility	09/18/2008
EMSL Analytical, Inc. (Libby, MT)	Asbestos Laboratory	09/16-17/2008
Hygeia Environmental, Inc. (Sierra Madre, CA)	Asbestos Laboratory	06/25-26/2008
EMSL Analytical, Inc. (Beltsville, MD)	Asbestos Laboratory	05/13-14/2008
EMSL Analytical, Inc. (Westmont, NJ)	Asbestos Laboratory	04/23-24/2008

[1] This laboratory is now located in Cinnaminson, NJ

5.1 On-site Audit Process

On-site audits are used by EPA to verify samples analyzed by their contract facilities are being processed in accordance with EPA requirements. Each on-site audit involves the general elements of preparation, on-site support, and report generation, which are modified as needed to fit the type of audit being performed. Preparation for asbestos laboratory audits typically involves ensuring the on-site audit checklist to be used is updated to reflect the latest methods and modifications required for Libby sample preparation and analysis; coordination with Region 8 to receive the most recent copies of the laboratory's SOPs, Quality Assurance Manual (QAM) and other needed documentation; and coordination with the EPA representative attending the audit with regard to travel logistics. If there are any anticipated problem areas based on prior evaluation of QA/QC data or validation reports, the auditor will discuss these with the EPA member of the audit team prior to the audit. The on-site audit generally starts with an entrance debriefing to the laboratory regarding what areas will be evaluated and the anticipated duration of the audit. This is followed by evaluating areas throughout the laboratory to verify adherence to Libby project analysis requirements, the laboratory preparation and analysis SOPs, and adherence to the requirements in the laboratory QAM. The areas typically audited in an asbestos laboratory include Sample Receipt, Log-in, Storage, and Chain-of-Custody (COC); Indirect and Direct Preparation of Samples; Transmission Electron Microscopy (TEM) Analysis; Polarized Light Microscopy (PLM) Analysis; and Quality Control and Quality Assurance. All laboratory staff involved with handling, preparing, analyzing, reporting, and performing QC on Libby samples are interviewed. Findings are recorded as identified, and reported to the laboratory at the exit debriefing. On-site audit reports detailing the findings are prepared and submitted to EPA typically within a month, and following EPA approval are sent to the laboratories, who are required to provide corrective action response to EPA regarding the findings. Areas in which findings are identified and evaluated during the next on-site audit to determine the degree to which laboratories have applied corrective action.

The results from the above-listed nine OU3 analytical support laboratory and two toxicology laboratory on-site audits are summarized below in the following categories:

- Deficiencies by Laboratory
- Laboratory Trends

- Deficiencies by Laboratory Process Area
- Laboratory Internal Audits
- Air Monitoring Samples
- Laboratory Responses
- Soil Preparation Facility (SPF) Audits
- Toxicology Laboratory On-site Audits

5.2 Deficiencies by Laboratory

A total of 112 observed deficiencies, compiled from the completed summary on-site audit reports, were identified from the nine OU3 laboratory on-site audits performed on five different laboratories between 2008 and 2012. Deficiencies from the two toxicology laboratory audits are not included in this total because these did not involve the preparation and/or analysis of asbestos samples. Of the nine laboratory audits, four were conducted in 2008, and five were performed in 2012. For four of the five laboratories audited in 2012, it was their second audit by EPA; one laboratory, (EMSL Analytical, Denver, CO) was a new laboratory audited for the first time in 2012. For all laboratory audits conducted in 2008 and 2012, an average of 12.4 deficiencies per audit was observed. The lowest number of deficiencies per audit was observed for EMSL Analytical (Westmont, NJ) in 2012 with seven (7), and the highest number of on-site deficiencies per audit occurred at Hygeia Environmental, Inc. (CA) in 2008 with 19. The deficiency totals, by laboratory, for all on-site audits conducted in 2008 and 2012 are provided in **Table 26**.

5.3 Laboratory Trends

A total of 34 deficiencies were identified in the four asbestos on-site laboratory audits performed during 2012 as compared to the 63 defects observed in the on-site audits of the same four laboratories in 2008. Note that five asbestos laboratory on-site audits were performed in 2012 (with 49 total defects observed) versus four on-site audits in 2008. The average of 8.5 defects per on-site audit in 2012 represents a 46.2% decrease from the 15.8 average number of defects per on-site audit (for the same four laboratories) recorded in 2008. All four laboratories audited in 2008 and again in 2012 showed a reduction in the number of defects, which suggests that all four laboratories applied corrective action in response to their initial audits in 2008.

The percent change (decrease or increase) in total defects from one on-site audit to the next can be a useful indicator of laboratory performance and/or applied corrective action. The percent change in defects between the four laboratories audited in 2008 to 2012 include Hygeia Environmental, Inc. (Sierra Madre, CA) (-47.4%), EMSL Analytical, Inc. (Westmont, NJ (-58.8%), EMSL Analytical, Inc. (Libby, MT) (-40.0%), and EMSL Analytical, Inc. (Beltsville, MD) (-33.3%).

Table 26 – Deficiencies by Laboratory (2008 - 2012)

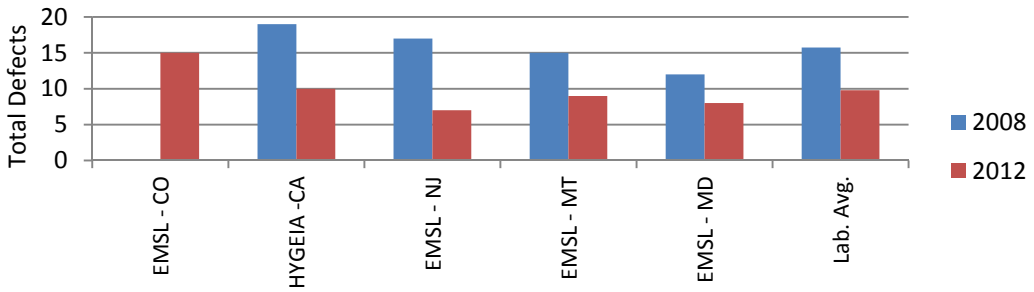
Laboratory	Deficiencies			Change In Defects per Audit	
	2008	2012	AVG	Increase/ (Decrease)	%Increase/ (%Decrease)
EMSL Analytical, Inc. (Denver, CO)		15	15	NA	NA
Hygeia Environmental, Inc. (Sierra Madre, CA)	19	10		(9)	(47.4%)
EMSL Analytical, Inc. (Westmont, NJ)	17	7	11.5	(10)	(58.8%)
EMSL Analytical, Inc. (Libby, MT)	15	9	12	(6)	(40.0%)
EMSL Analytical, Inc. (Beltsville, MD)	12	8	10	(4)	(33.3%)

Table 26 – Deficiencies by Laboratory (2008 - 2012)

Laboratory	Deficiencies			Change In Defects per Audit	
	2008	2012	AVG	Increase/ (Decrease)	%Increase/ (%Decrease)
Total	**63**	**49**			
Average	**15.8**	**9.8**		**(7.3)**	**(44.9%)**

Figure 1 provides a graphic of the number of defects by laboratory of all on-site audits performed in 2008 and 2012.

Figure 1 – Asbestos On-site Audit Trends: Total Defects by Laboratory (2008-2012)



5.4 Deficiencies by Laboratory Process Area

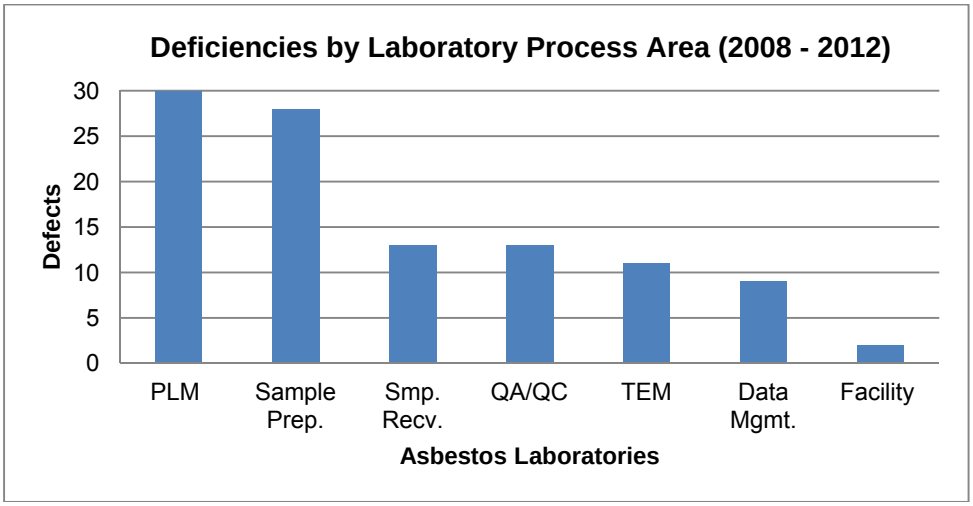
The 112 asbestos on-site audit deficiencies identified in the nine on-site laboratory audits performed in 2008 and 2012 were trended by eight laboratory process areas. The laboratory process categories in which the majority of the observed deficiencies occurred include PLM, Sample Preparation, Sample Receiving, and QC/QA. Categories with the least frequently occurring deficiencies included TEM, Facility, and Data Management.

Table 27 and **Figure 2** show the laboratory process categories evaluated, the number of deficiencies observed in each from the combined 2008 and 2012 on-site audits, and the percentage of deficiencies observed by category.

Table 27 - OU3 On-site Laboratory Audit Deficiencies by Laboratory Process Area - 2008 to 2012

Laboratory Area	Deficiencies	Percentage
PLM Analysis	30	28.3%
Sample Preparation	28	26.4%
Sample Receiving	13	12.3%
QC/QA	13	12.3%
TEM Analysis	11	10.4%
Data Management	9	8.5%
Facility	2	1.9%
Total	**106**	**100%**

Figure 2 – Asbestos On-site Audit Trends: Deficiencies by Laboratory Area (2008 - 2012)



Examples of high frequency deficiencies by laboratory process category that were observed in the five on-site audits performed in 2012 are summarized as follows:

Polarized Light Microscopy (PLM) Analysis - In four of the five laboratories audited in 2012, the Laboratory Duplicate Cross-check (LDC) optical property observations were being recorded on the same bench sheet as the observations for the original (first) analysis, and therefore were not "blind". In three of the laboratories, the procedure used for performing the PLM analysis of finely ground soil samples did not comply with the procedure described in SOP SRC-Libby-03 in that suspect fibers were picked out after rather than prior to preparing the five random slide mounts. In three of laboratories, the permanently mounted, LA reference slides of 0.2% and 1.0% were not prepared "in-house," but by one of the other Libby laboratories.

Indirect and Direct Preparation of Air Filter and Dust Samples - Three of the five laboratories audited in 2012 had deficiencies related to balance calibrations, including failure to perform daily calibration checks of balances used to weigh samples and the use of an outside service for annual balance calibrations. In two of the laboratories, the Effective Filtration Area (EFA) of the disposable filter assembly was not being determined for each lot of filters received.

Sample Receipt, Storage, Log-in, and COC - Two of the five laboratories audited in 2012 did not have HEPA hoods in the sample receiving area or the hoods were not properly identified to allow flow checks and HEPA filter changes to be documented.

Quality Control and Quality Assurance – Several of the laboratories audited in 2012 did not always implement or maintain adequate quality systems. EMSL-MT did not perform internal audits at the required frequency, EMSL-NJ did not have an adequate quality system in place to track corrective actions, and Hygeia did not control obsolete documents including QAMs and SOPs. Two laboratories did not adequately perform air monitoring including EMSL-MD which did not always perform cleanups and re-sampling in response to internally or externally-identified asbestos contamination, and Hygeia which did not perform air monitoring of the analytical areas at the frequency described in their written procedures.

Transmission Electron Microscopy (TEM) Analysis - There were no common deficiencies in TEM analysis identified in the 2012 on-site audits. One laboratory had no defects in this

category, while the other four each had one deficiency related to documentation, availability of lab modification, or the availability of instrument LA reference spectra.

Data Management - There were no common deficiencies in Data Management identified in the 2012 on-site audits. Three of the five laboratories had no deficiencies in this category. Deficiencies related to record-keeping and availability of all required procedures were observed in the EMSL-Beltsville and EMSL-Denver audits.

Facilities - There were no facility-related deficiencies identified in the 2012 on-site audits.

5.5 Laboratory Responses to On-site Laboratory Audits

EPA requires that laboratories provide responses to on-site audit reports which include the laboratory's proposed corrective action to each of the identified findings. These laboratory responses assist EPA in "closing the loop" on laboratory deficiencies, and help resolve method interpretation issues. Laboratory responses to all reports for the 2008 and 2012 on-site audits have been received from the five OU3 support laboratories. All laboratories provided proposed corrective actions for the identified findings along with objective evidence as applicable. No findings were contested. These laboratory-proposed corrective actions in response to the 2012 audits will be verified during the next round of scheduled audits. As shown in **Table 26**, the total number of findings by the OU3 support laboratories decreased between 2008 and 2012 from 58.8% to 33.3%, which suggests that corrective action has been performed in response to previous audit findings.

5.6 Soil Preparation Facility (SPF) Audits

In 2008 and 2012 QATS supported three soil preparation facility on-site audits. The ESAT Region 8 SPF in Troy, MT and the CDM CSF in Denver, CO were audited in 2008, and a follow up audit of the ESAT Region 8 SPF was performed in 2012. A summary of deficiencies from the three on-site audits are listed in **Table 29.**

5.6.1 Deficiencies by Laboratory

A total of 35 observed deficiencies, compiled from the completed summary on-site audit reports, were identified from the three soil preparation facility on-site audits performed on two different facilities in 2008 and 2012. Of the three audits, two were conducted in 2008 and one in 2012. For all three audits, an average of 11.7 deficiencies per audit was observed. The lowest number of deficiencies per audit was observed for ESAT Region 8 SPF in 2008 with eight (8), and the highest number of on-site deficiencies per audit occurred with CDM CSF in 2009 with 17. The deficiency totals, by facility are provided in **Table 29**.

Table 29 – Deficiencies by Facility (2008 and 2012)

	Deficiencies			Change In Defects per Audit	
Soil Preparation Facility	2008	2012	AVG	Increase/ (Decrease)	%Increase/ (%Decrease)
CDM Close Support Facility (Denver, CO)	17		17	NA	NA
ESAT Region 8 Soil Prep. Facility (Troy, MT)	8	10	9	2	25.0%
Total	**25**	**10**			
Average	**12.5**	**10**		**(2.5)**	**(20.0%)**

5.6.2 Laboratory Trends

Ten deficiencies were identified in the ESAT Region 8 SPF on-site audit performed in 2012 as compared to the eight defects observed at the same facility in 2008. This represents a 25.0% increase from the 2008 audit. This increase is due mainly to one additional deficiency in the bulk drying, grinding and splitting, QA/QC, sample receiving, and sieving categories. Decreases were observed in the facility, sample preparation, and health and safety categories.

The 35 on-site audit deficiencies identified in the three soil preparation facility on-site audits were trended by eight laboratory process areas. The laboratory process categories in which the majority of the observed deficiencies occurred include Bulk Drying, Grinding and Splitting, QC/QA, and Sample Receiving. Categories with the least frequently occurring deficiencies included Facility Sieving of Preparation Samples, Sample Preparation, and Health and Safety.

Table 30 shows the laboratory process categories evaluated, the number of deficiencies observed in each from the combined 2008 and 2012 on-site audits, and the percentage of deficiencies observed by category.

Table 30 - OU3 On-site Laboratory Audit Deficiencies by Laboratory Process Area - 2008 to 2012

	Deficiencies				
Laboratory Area	**SPF 2012**	**SPF 2008**	**CSF 2008**	**Total**	**Percentage**
Bulk Drying	3	2	6	11	31.4%
Grinding and Splitting	3	2	1	6	17.1%
QC/QA	2	1	3	6	17.1%
Sample Receiving	1	0	4	5	14.3%
Facility	0	1	2	3	8.6%
Sieving of Preparation Samples	1	0	1	2	5.7%
Sample Preparation	0	1	0	1	2.9%
Health and Safety	0	1	0	1	2.9%
Total	**10**	**8**	**17**	**35**	**100%**

5.7 Toxicology Laboratory On-site Audits

In 2011-2012 QATS supported the on-site laboratory audits of two toxicology laboratories, one at the Oregon State University (OSU) Aquatic Toxicology Laboratory in Albany, OR, and the other at Fort Environmental Laboratories, Inc. (FEL), in Stillwater, OK. A summary of deficiencies from the two on-site audits are listed below:

- The audit of the OSU Aquatic Toxicology Laboratory involved an evaluation of the pilot study protocol, *ASB-RBT-AS5d-001 (Revision 4) – Evaluation of Free-fiber Libby Amphibole Asbestos Toxicity in Laboratory Water to the Rainbow Trout (Oncorhynchusmykiss)* against the procedures used by the laboratory for all aspects of the study. These included the shipping and receiving of test organisms, standards, and collected samples; the preparation and monitoring (physical and chemical) of test chambers; sample collection; a review of the laboratory's record keeping practices for shipping and receiving, test chamber preparation, and analytical measurements; the availability of written procedures; and the presence of a viable quality assurance/quality control (QA/QC) program. Several on-site audit observations were identified, including

the standards used were not received under proper chain-of-custody (COC) and stored in a secure location within the facility; collected samples were not shipped as scheduled; use of an inadequate COC form; failure to always document adjustments made to the water quality; use of insufficiently specific toxicology forms; deviations from protocol were not communicated to the proper authority; the size and type of filter used to collect samples were not recorded; and the QA/QC program implemented was not as described in the laboratory Quality Assurance Plan.

- The audit of the Fort Environmental Laboratories, Inc. (FEL) involved an evaluation of the toxicological study to examine the effects of LA on the complete metamorphosis of ranid amphibians. The results of the study will be used to support the evaluation of potential ecological risk at the Libby Asbestos Superfund Site in Libby, Montana. This study involved a complete amphibian metamorphosis assay in which ranid larvae, beginning at Gosner Stage 20, were exposed to LA. The general experimental design entailed exposing the test organisms to contaminated soil collected from the Libby Superfund Site and two controls, inert sand and reference sediment. The primary endpoints of the study were survival, developmental stage, time to metamorphosis for each test organism, median time to metamorphosis for each replicate, metamorphic count, external and internal malformations, whole body weight, and snout-vent length of each surviving test organism. The on-site evaluation identified only one observation: There was a failure to analyze the laboratory control (inert sand) and reference sediment for the presence of LA prior to initiation of the in-life study.

5.8 Laboratory Internal Audits

Each laboratory conducts internal audits of their specific operations on an annual basis using appropriate checklists. During the on-site audits, the Audit Team reviews with the laboratory staff any significant findings noted in their internal audit reports. **Table 28** shows the internal audit history from 2007 to 2012 of the five laboratories that currently provide support to OU3 investigation activities. Internal audits were performed by two of the five laboratories in 2007, by all of the laboratories between 2008 and 2011, and by four of the five laboratories in 2012. **Table 28** below lists the dates of the laboratory internal audits between 2007 and 2012:

Table 28 - Internal Audit History for OU3 Support Laboratories (2007 - 2012)

Laboratory	2007	2008	2009	2010	2011	2012
EMSL-Westmont	None	4/29/2008	4/13-15/2009	4/27-29/2010	7/7-8/2011	None
EMSL-Denver	None	2/25/2008	5/18/2009	3/31/2010	3/23-25/2011	10/30/2012
EMSL-Beltsville	None	11/12-14/2008	12/7-9/2009	12-21-22/10	12/15-16/2011	12/19-20/12
EMSL-Libby	6/24/2007	9/10/2008	10/13-14/09	1/14-15/2010	1/12/2011	9/24-25/2012
Hygeia	8/20/2007	9/3-4/2008	11/13/2009	7/14-15/2010	8/15-16/2011	9/24-28/2012

5.9 Air Monitoring Samples

An environmental contamination monitoring program is required at each laboratory that analyzes samples from Libby. Specifics regarding the requirements of the laboratory monitoring program for each laboratory are described in the laboratory QA management plan. The laboratory QAM should immediately contact the LC and the QATS contractor of any laboratory contamination monitoring results that are outside of the appropriate acceptance criteria. From April 20, 2010 through December 12, 2012 112 air monitoring samples were collected at the EMSL Laboratory in Libby, Montana. Samples were collected at various locations throughout

the laboratory including, the transmission electron microscope laboratory, the polarized light microscopy laboratory, the base, and the reception area. Of the 112 air monitoring samples collected, two had results that exceeded the acceptance criteria, as described below:

- July 20, 2012 – An air monitoring sample collected from the PLM room on this date contained 1 LA structure. Upon being notified of this contamination, which was minor, the appropriate corrective action was initiated. The area was re-cleaned and re-sampled to ensure the problem had been addressed.

- September 20, 2012 – An air monitoring sample collected in one of the TEM rooms on this date contained 2 chrysotile structures. Corrective action was taken by the laboratory; however since CH is not an analyte of interest, no additional action was necessary.

6.0 Laboratory Mentoring Program

In 2012 EPA requested the support of QATS to mentor Materials Analytical Services (MAS) in Suwannee, GA to perform asbestos analysis of samples collected from Libby OU3. Mentoring activities included a review of project-specific procedures (i.e. SOPs, SAP Summaries and Modifications) and electronic data deliverable tools, and also the selection of previously analyzed air, water, and soil samples, which were shipped to MAS for asbestos analysis by PLM and TEM methodologies. Upon completion of the analyses, QATS performed a review of both the hardcopy and electronic deliverables to ensure completeness and compliance with project-specific requirements. The mentoring process continued into September, during which time the laboratory demonstrated proficiency in both PLM and TEM analyses. On September 24, 2012 QATS submitted the first of two technical memos summarizing the mentoring activities associated with the PLM and TEM analyses by MAS of both soil and air QC samples which included re-preparations and inter-laboratory analyses. QATS was in the process of reviewing the TEM water deliverables when a perceived conflict of interest (COI) between MAS and the Primary Responsible Party (PRP), WR Grace, was identified by EPA. EPA directed QATS at this point to terminate all mentoring activities.

7.0 Development and Review of Standard Operating Procedures (SOPs) and Other Quality Documents

7.1 Standard Operating Procedures (SOPs)

The following SOPs were developed for the data validation of TEM and PLM data by the multiple methods used at the Libby site:

a) SOP QATS-70-094-00 (Standard Operating Procedure for the Validation of Libby Polarized Light Microscopy (PLM) Data Deliverables)

b) SOP QATS-70-095-00 (Standard Operating Procedure for the Validation of Libby Transmission Electron Microscopy (TEM) Data Deliverables)

7.2 Laboratory Modifications

During the period QATS supported the development or revision of sixteen (16) project-specific laboratory modifications. Listed below are summary descriptions and revision dates of each laboratory modification.

a) LB-000016H (Revised 9/25/2012) – This modification documents permanent modifications and clarifications to TEM structure recording rules for ISO 10312 and documents previous historical modifications and clarifications. This modification applies to all Libby TEM samples where the ISO 10312 counting rules apply, regardless of sample matrix (air, dust, water, woodchip/duff, tree bark, and tissue samples).

b) LB-000020B (Revised 3/19/2012) – This modification applies to the preparation and analysis of water samples for the Libby Project. As of 07/27/2010, it requires all water samples associated with the Libby Superfund Site (including OU3) to undergo treatment with ozone/UV light and sonication prior to filtration as specified in Section 6.2 of EPA Method 100.1 (EPA 1983a). Only polycarbonate (PC) or mixed cellulose ester (MCE) filters with a pore size of 0.2 µm or smaller should be used for filtering water samples. On the bench sheets, the preparation date should be recorded as the filtration date, not the grid preparation date. Recording rules will be as described in the ISO 10312 (ISO 1995) method, except that the aspect ratio and minimum length requirements will be specified in the applicable governing Analytical Requirements Summary Sheet. The reason for the modification is studies performed by the EPA (EPA 1983b) have indicated that water samples collected for asbestos concentration determination that are not completely sterile are complicated by microbial growth which leads to the potential for clumping of fibers within organic matter and clumps adhering to the sides of the vessel. This has a two-fold effect towards underestimating asbestos estimation; fibers within clumps of organic matter cannot be adequately identified by microscopy and fibers adhering to the sides of the vessel decrease the fiber concentration in the water.

c) LB-000029D (Revised 3/12/2013) – This modification provides permanent clarifications to laboratory-based quality control (QC) analysis requirements for TEM. The purpose is to standardize the frequency of analysis and procedures for the selection and interpretation of the results for laboratory-based TEM QC analyses (regardless of sample medium).

d) LB-000055B (Revised 11/2/2012) – This purpose of this laboratory modification is to address sample collection procedures for the Outdoor Ambient Air Monitoring Programs for the Libby Asbestos Superfund Site, including the ambient air programs for Operable Unit 4 (OU4) and OU7 (Troy). Due to meteorological conditions prevalent in Libby in the late fall (e.g., fog, inversions, other potential precipitation), the collected air filters have the potential to arrive at the laboratory in a damp condition. To allow these samples to be properly prepared for TEM analysis and to prevent subsequent biological growth, this modification requires all ambient air samples to be dried upon receipt at the on-site laboratory (e.g., EMSL-Libby), prior to further preparation/analysis at the on-site laboratory, or prior to transfer to another laboratory for further preparation/analysis.

e) LB-000066C (Revised 9/11/2007) – This temporary modification applies to all investigative samples (as defined in the most recent version of LB-000053) evaluated at the Libby Superfund Site. This temporary modification requires all analytical laboratories to: 1) complete the structure comment field to characterize particles with regard to the levels (presence/absence) of the sodium and potassium peaks observed in the energy dispersive spectrometry (EDS) spectrum; 2) record on the data sheets all non-asbestos material (NAM) particles that are “close calls”; 3) increase the frequency that EDS spectra are saved for “LA” and “close call” structures; 4) increase the frequency that photographic images of particle morphology are recorded for “LA” and “close call”

structures; and 5) complete the comment field to record mineral type of each recorded particle, including “LA”, “OA”, “C” and “close call” NAM particles.

Note: Modification LB-000066C is only to be used if specified.

f) LB-000066D (Revised 7/2/2010) – This permanent modification applies to all Libby site investigative samples as defined by in the relevant SAPs and analyzed by TEM. This modification does not apply to non-investigative samples. Based on this modification, all analytical laboratories shall: 1) indicate on the count sheet the presence or absence of sodium and potassium in all recorded structures (except chrysotile); 2) record on the count sheet “close-call” NAM particles; 3) record the probable mineral species of each recorded structure; 4) record EDS spectra of “LA” and “close-call” NAM particles; and 5) record 1 photomicrograph of a SAED (selected area electron diffraction) pattern for each “LA” or “OA” amphibole type encountered in a sample.

g) LB-000067C (Revised 4/1/2013) - This modification provides direction on how to improve consistency in the recording and reporting of structures for all TEM methods for the Libby Project. It also consolidates the three modifications applicable to all TEM methods into a single modification.

h) LB-000073C (Revised 12/6/2012) - This modification provides permanent clarifications to inter-laboratory analyses for the Libby-specific PLM-VE (SRC-LIBBY-03) and PLM-Gravimetric (SRC-LIBBY-01) methods; and standardizes the selection and analysis procedures for inter-laboratory samples of soil.

i) LB-000085A (Revised 5/4/2012) – The purpose of this modification is to standardize the frequencies and performance criteria of instrument calibrations at all TEM laboratories that analyze samples for the Libby Project. Contamination monitoring by air sampling at the labs is also described in this modification.

j) LB-000087 (Revised 5/4/2012) – This modification documents clarification of the PLM NIOSH Method 9002 asbestos mineral identification criteria as applied to the identification of tremolite-actinolite, and its presence as “LA” in soils collected from the Libby Superfund Site. It also describes the historical recording and reporting of tremolite-actinolite and “LA”, respectively, in samples analyzed by NIOSH Method 9002 prior to 03/14/2012; how the Scribe database will be updated to address the described inconsistencies; and how samples identified as containing tremolite-actinolite by this method will be qualified to document their inclusion in “LA” solid solution series in all future deliverables.

k) LB-000088 (Revised 12/17/2012) – This modification documents the effective dates on which the project soil preparation facility (SPF) and analytical laboratories are to adhere to SOPs ISSI-Libby-01, SRC-Libby-03, and SRD-Libby-01 when performing PLM-VE, Gravimetric analysis, or particle size reduction.

8.0 Conclusions & Recommendations

Although several quality issues were identified from the review of the field and laboratory QC results, data validation results, and on-site audit results; overall, the QC data evaluated from the assessments appear to be of good quality and capable of supporting risk assessment and other

decision making related to the results of the associated samples. A summary of the issues identified by QC category along with recommendations are provided as follows:

Field QC

Field QC includes field, lot, and rinsate blanks; and field duplicates. Four field issues were identified:

- Field blanks, duplicate blanks were not always collected at the frequencies described in the applicable SAPs.
- Lot blanks have not been collected since 2010 and it is unknown whether the lots checked in 2010 are still in use.
- Libby Amphibole (LA) was identified in two field blanks.
- Field duplicate pairs with Poisson ratio rates outside of the desired 95% confidence interval were detected.

To address the above issues, it is recommended that field SAPs be read and acknowledged by all field personnel, and that COCs are reviewed to ensure that field QC are collected at the frequencies required by the investigation-specific SAPs. To ensure that the filters used to collect air samples are from the lots checked in 2010, the applicable FSDSs should be checked to ensure that the filter lot numbers recorded match those that have been verified.

Regarding the field blanks, contamination was limited to a single LA structure detected in two out of the 86 field blanks collected. As a result of this contamination, all associated samples with positive LA concentrations will be qualified “FB” to warn users of the potential for cross contamination. It should also be noted that LA fibers were also detected in an additional two field blanks, but that these particular samples are part of an investigation concerning the possibility that samples were either mixed up in the field or misidentified at the laboratory. For the field duplicates, sample pairs falling outside the desired confidence interval were limited to water, tree bark, and duff samples suggesting that reproducibility is difficult due to inherent sampling variability associated with the collection of these media types.

Laboratory QC

Laboratory QC includes both intra- and inter-laboratory results. Two laboratory QC issues were observed:

- Intra-laboratory QC analysis frequency was not always performed at the project level or specific to samples collected from OU3.
- The inter-laboratory results for TEM did not always fall within the desired accuracy parameters.

The Intra-laboratory quality control analyses includes laboratory blanks, recount same, recount different and verified analyses. The laboratories, for reasons not determined, ceased tracking intra-laboratory QC these analyses separately for OU3 and instead tracked them with samples collected from other operable units. This issue was brought to the attention of the applicable laboratories who have since resumed assigning intra-laboratory analyses at the project level. In order to correct this deficiency, QATS would recommend that the laboratories perform these QC analyses retroactively in order to meet the requirements specified in each of the investigative-specific SAPs.

As noted in Tables 14A-14E in Section 3.3.1.4 of this report, the results from the inter-laboratory results failed to meet the criteria established for structure length, structure width and structures per GO. Although some of these discrepancies can be attributed to differences in instrumentation, analytical experience, and the nature of the structures themselves, which can exhibit different elemental compositions at different points along the fiber (i.e. transitional fibers), the identified discrepancies highlight the need for further discussion among the participating laboratories. This would help to achieve greater consistency between laboratories when identifying samples as either "close call" or NAMs, and also help them gain a better understanding of the length and width discrepancies observed. Increasing the frequency of inter-laboratory analyses for specific media should also be considered.

Data Validation

Data validation for asbestos in ambient air, tree bark, mine waste, surface water, duff, sediment, forest soil, groundwater, and pore water was performed in accordance with the applicable methods, SAP Analytical Requirements Summaries, Laboratory Modifications, and CB&I - QATS Libby-specific data validation SOPs. Selection of five percent (5%) of the sample results to validate was performed by randomly choosing sample results by laboratory, method, and media. A total of 360 field samples from 30 Laboratory Job Numbers, analyzed by five different laboratories between 2007 and 2012, were selected for validation. In addition to the 360 field samples validated, 43 blanks and 25 QC samples were validated.

Very few OU3 asbestos results were qualified. Qualifiers were applied to only one field sample result and one QC sample result (recount different) of the 360 asbestos sample results validated (0.56%); 99.4% of the OU3 asbestos results for samples analyzed between 2007 and 2012 required no qualification. The two sample results qualified were due to the failure of the laboratory to perform and/or document daily calibration activities. The lack of recording instrument calibrations (i.e. daily TEM alignment and energy checks) has been addressed though the initiation of laboratory modification LB-000085A, which requires the laboratories to provide instrument calibration on a quarterly basis.

Discrepancies were observed in the bench sheet/EDD information comparisons due to information omissions and typographical errors, which were reported in the EDD/Bench Sheet Discrepancy Table in the Asbestos Validation Summary Reports. The discrepancies ranged from minor (i.e., typographical errors in fields that do not affect the sample results) to more severe discrepancies (i.e., typographical errors for fiber length and/or width; primary filter area; and mineral identification which could affect the sample result, or date analyzed discrepancies which could affect the daily calibration verification). A total of 89 of the 360 sample results validated (24.7%) contained some type of bench sheet/EDD discrepancy. However, 86 of these 89 (97%) were minor typographical discrepancies.

On-site Audits

Fourteen (14) on-site audits were performed, nine of which were asbestos laboratory audits, three were soil preparation facility audits, and two that were asbestos toxicology study laboratory audits. For the asbestos laboratory audits, the areas audited in an asbestos laboratory include Sample Receipt, Storage, Log-in, and Chain-of-Custody (COC); Indirect and Direct Preparation of Samples; Transmission Electron Microscopy (TEM) Analysis; Polarized Light Microscopy (PLM) Analysis; and Quality Control and Quality Assurance.

A total of 112 observed deficiencies were identified from the nine OU3 laboratory on-site audits performed on five different laboratories between 2008 and 2012. Overall a 46.2% decrease in the average number of defects per on-site audit (for the same four laboratories) recorded in 2008 was observed in 2012. All four laboratories audited in 2008 and again in 2012 showed a reduction in the number of defects, which suggests that all four laboratories applied corrective action in response to their first audits in 2008. Of the five asbestos laboratory audits in 2012, the first-time audit of EMSL-CO had the most defects with 15, while EMSL-NJ had the fewest number of defects with seven. Laboratory responses to all reports for the 2008 and 2012 on-site audits have been received from the five OU3 support laboratories. All laboratories provided proposed corrective actions for the identified findings along with objective evidence as applicable. No findings were contested. These laboratory-proposed corrective actions in response to the 2012 audits will be verified during the next round of scheduled audits.

For the ESAT Region 8 SPF audits, there was a slight increase (from 8 to 10) in the number of deficiencies observed in the 2012 on-site audit versus the audit in 2008. This increase could partially be attributed to the presence of several new staff in 2012 who were not present during the 2008 audit.

A final recommendation is that QC activities (i.e. on-site audits, validation and inter-labs) be scheduled on an annual basis in order to identify and resolve quality issues in a timely manner, which will minimize the impact on the quality of associated data.

Attachment 1

EMSL Analytical (Libby, MT) Issues and Concerns

CB&I
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Las Vegas, NV 89120
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Memorandum

From: Michael P. Lenkauskas
Date: August 30, 2013
Subject: EMSL Analytical (Libby, MT) Issues and Concerns

The following is a summary of issues and analytical data discrepancies associated with the EMSL Analytical laboratory in Libby, Montana. The discrepancies, identified by CDM Smith and CB&I, raise concerns about the quality of the data provided by this laboratory for samples collected from Operable Unit 3 (OU3) of the Libby Superfund Site. The specific issues include:

- TEM Inter-lab sample preparation issues
- Inadequate frequency of project-specific QC analyses
- Possible misidentification of samples
- Result discrepancies between TEM rapid TAT and full analysis of OU3 water samples

TEM Inter-lab Sample Preparation Issues

The EMSL Analytical laboratory experienced an unusually high percentage (37%) of damaged grid openings (GOs) on re-preparations prepared for the 2010 and 2011 TEM inter-laboratory, which resulted in these samples having to be re-prepped again, slowing down what turned out to be an already laborious process. Upon identification of this issue on March 25, 2013, the laboratory was directed to investigate and apply the necessary correction actions prior to preparing the re-preparations for the 2012 TEM inter-laboratory study about to be initiated. The root cause of the damaged GOs, as determined by the laboratory, is described in the attached corrective action (CAR# 1303-1), was the following:

- Grid opening size, EMSL uses a custom made grid with an opening of 0.0130 sq. mm;
- Grid condition;
- Carbon coating thickness;
- Ash time; and
- Packaging and shipping.

In addition to using a grid with a smaller grid opening size (15x15 grids with a G.O.A. of 0.0064 sq. mm) the laboratory is now also pre-cleaning the grids and has adjusted the asher and carbon coating settings. Since none of the 11 samples re-prepped by the laboratory for the 2012 TEM inter-laboratory were received damaged, the corrective actions initiated by the laboratory appear to have resolved the issue.

It should also be noted that undissolved filter material has also been observed on EMSL grid preparations, which will be investigated on a laboratory-by-laboratory basis during the 2012 laboratory on-site audits.

Inadequate Frequency of Project-specific QC Analyses

A review of the QC analyses available in the OU3 database for samples analyzed in 2012 revealed that the frequency at which QC analyses were performed for both TEM and PLM analyses during this period was not in accordance with the criteria described in Laboratory Modification LB-000029D and SOP SRC-Libby-03 (rev. 3) for TEM and PLM, respectively. The following table provides a summary of analyses

performed, the required frequency, the number of QC analyses that should have been performed, and the actually number and percentage of QC analyses that were performed:

Method	QC Type	Sample Analyses	Required Frequency	Performed	Actual Frequency
TEM	LB	293	4%	7	2.4%
TEM	RS	293	1%	0	0%
TEM	RD	293	2.5%	3	1%
TEM	VA	293	1%	2	0.7%
TEM	RP	293	1%	5	1.7%
PLM	LDC	65	8%	1	1.5%
PLM	LDS	65	2%	3	4.6%

Although QC analyses were not performed at the required frequency on a project-specific basis (OU3), they were prepared at the required frequency for all of the operable units combined. This discrepancy was brought to the attention of EMSL Analytical Management on May 22, 2013, who performed an investigation and determined that separate QC logbooks were maintained up until June 25, 2012, at which time they were combined[1]. Effective May 23, 2013 samples received from OU3 are once again recorded in a separate, OU3-specific, QC logbook, ensuring that project-specific QC will be performed at the required frequencies.

Possible Misidentification of Samples

A review of the results from surface water samples collected from OU3 during the spring of 2012 and analyzed by the laboratory indicates that samples were misidentified either in the field during collection or in the laboratory while being processed. Samples possibly misidentified are summarized in the following table:

Index ID	Sample Type	Date Prepared	Date Analyzed	Structures	Comments
P5-10013	Field Sample	5/19/12	5/25/12	0	Same preparation batch. Lab indicated sample P5-10014 was cloudy.
P5-10014	Field Blank		5/26/12	25	
P5-10067	Field Sample	6/20/12	6/26/12	25	Field sample/field duplicate pair
P5-10068	Field Duplicate		6/26/12	1	
P5-20018	Field Sample	5/17/12	6/01/12	0	Field sample/field duplicate pair. Lab RP had 50 structures.
P5-20019	Field Duplicate		6/02/12	65	
P5-20085	Field Sample	7/04/12	7/09/12	5	Field sample/field duplicate pair
P5-20087	Field Duplicate		7/09/12	27	
P5-20225	Field Sample	9/20/12	11/08/12	25	Field sample/field duplicate pair
P5-20226	Field Duplicate		11/08/12	62	

Although sometimes analyzed on separate days, each of the sample pairs in question were prepared on the same days by the same preparer, increasing the possibility that the misidentification of at least the field duplicate pairs at the laboratory. It should also be noted that with the exception of the sample pair prepared and analyzed in September and November, respectively, which has results that may or may not indicate the samples were misidentified, the remaining samples, which exhibit much greater disparity, were all prepared and analyzed during the spring/early summer 2012.

The potential that the misidentification of samples was brought to EMSL Analytical Management's attention, and on February 19, 2013 the laboratory provided a memo to both EPA and Remedium summarizing the findings of their investigation. The first section of this memo discusses the TEM Rapid TAT versus TEM full analysis discrepancies, which are discussed below. Concerning the possible misidentification of samples, the laboratory offered the explanation that at the time of the misidentifications the laboratory was operating beyond its capacity, creating a disorganized environment

[1] Note that this timeframe coincides with the change in the OU3 laboratory subcontracting mechanism from Remedium to TechLaw.

with staff trying to handle too many responsibilities. Procedural changes put in place by the laboratory to prevent similar situations for occurring in the future include:

- Expansion of the sample preparation area creating a less cluttered workspace in which to stage more samples in an organized manner
- Restricting the number of jobs being prepared simultaneously
- Having one individual track the progress of each individual lab job
- Provide training and improve intra-laboratory communication to better handle lab capacity issues

Note: Although this memorandum indicated that the capabilities of the Denver laboratory were to be increased to handle duff and water samples, as of the spring of 2013, this action has not been implemented.

Result discrepancies between TEM rapid TAT and full analysis of OU3 water samples

For a subset of the Kootenai River water samples collected in 2012, the EMSL-Libby laboratory was requested to perform a "rapid" TAT analysis. This analysis was performed using the same preparation techniques and counting rules as the traditional "full" analysis, but only required the analyst to record the total number of countable LA structures per GO (i.e., recording of structure-specific attributes, such as length, width, and structure type, was not required) to facilitate the faster reporting of water concentrations. Following the rapid TAT analysis, each water sample was subsequently re-analyzed[2] using the traditional full analysis reporting requirements.

A comparison of the rapid vs. full analysis results performed in January/February 2013 revealed significant discrepancies between the reported water concentrations for several samples (examples provided below):

Index ID	Total LA Water Conc. (MFL)	
	Rapid Analysis	Full Analysis
P5-10004	3.7	0
P5-10010	97	0
P5-10008	62	0
P5-10013	40	0

These discrepant results were brought to EMSL Analytical Management's attention, and the laboratory repeated the rapid and full analysis for a subset of the Kootenai River water samples (from the raw water that was in archive) to identify the nature of these discrepancies. The results of these repeated analyses indicated that the reported water concentrations from the original rapid analysis were not confirmed, but that the original full analysis results were confirmed *for most samples*. On this basis, the laboratory provided a memo to both EPA and Remedium on February 19, 2013, recommending that "*all rapid results should be disregarded in favor of the full ISO analyses*". This memo did not specify the reason for the differences between the rapid and full analysis results, but EMSL later noted that the analyst performing the rapid analysis may have utilized PCM recording rules, resulting in the recording of diatom fragments as countable structures.

However, as noted above, the repeat full analyses did not confirm the results for all samples. In particular, for a subset of samples listed below, the repeat full analysis did not confirm either the original rapid analysis or the full analysis:

Index ID	Total LA Water Conc. (MFL)		
	Original Analysis		Repeat
	Rapid Analysis	Full Analysis	Full Analysis
P5-10018	78	35	0
P5-10017	37	58	0

[2] Because the grids from the rapid analysis were often blown due to the original examination, this re-analysis was performed using a newly prepared set of grids from the original filter.

Index ID	Total LA Water Conc. (MFL)		
	Original Analysis		Repeat
	Rapid Analysis	Full Analysis	Full Analysis
P5-10015	34	60	0

In the case of one field blank (P5-10014), the re-analysis supported the unexpected results of the original full analysis, which reported a total LA water concentration of about 25 MFL. However, EMSL is looking further into the possibility that there were clip and archive grid location mix-ups that occurred at EMSL-Libby that may explain these unexpected results.

Because of these discrepancies, the validity of the original full analysis results is also uncertain.

Resolution of discrepancies for OU3 water samples

Re-analyses of samples collected in 2012

As a consequence of the discrepancies discussed above, several re-analyses were performed of the water samples collected in 2012 from the Phase V Part A (Kootenai) and Part B (Ecological) studies to confirm the originally reported results. This re-analysis effort included the analysis of a subset of water samples from the Kootenai River study (i.e., samples collected during Rounds 1 through 5 from stations LRC-6 and UKR-0) and the in-stream fish toxicity tests (i.e., a subset of the LRC surface water samples from the eyed egg study and 20% of the surface water samples from the fry study). These re-analyses were performed by EMSL-Cinnaminson in July/August 2013 from the raw water[3].

The following table summarizes these results. As shown, of the 24 samples that were re-analyzed, there were 8 samples where the repreparation analysis performed by EMSL-Cinnaminson was statistically different from the original analysis performed by EMSL-Libby (based on a Poisson ratio comparison test at a 90% confidence interval). This means that the difference in LA water concentrations between the original analysis and the repreparation analysis was more than can be attributed to Poisson counting error alone. For the 3 samples that were different from the Part A program (Kootenai), these results confirmed that some type of filter mix-up had occurred for samples P5-10015, P5-10017, and P5-10018 during the original analysis at EMSL-Libby. For the other 5 samples that were different from the Part B program (Ecological), there appears to be a consistent bias, with EMSL-Cinnaminson reporting higher concentrations than EMSL-Libby. Although for most of these samples, the concentrations are usually within a factor of about 3, there was one sample (P5-20027) where the reported concentration by EMSL-Cinnaminson is about 90 times higher than what was reported by EMSL-Libby, which may indicate another potential filter mix-up.

Re-analyses of samples collected in 2013

In addition, approximately 20% of the water samples collected as part of the 2013 eyed egg study were also be randomly selected *a priori* for re-analysis by EMSL-Cinnaminson in July/August 2013. These re-analyses were performed from either the originally prepared filter or the raw water (depending upon the nature of the archived sample).

The following table summarizes these results. A total of 17 samples were selected for re-analysis by EMSL-Cinnaminson; 10 samples were reprepared from the filter (filter was prepared by EMSL-Libby) and 7 samples were reprepared from the raw water. As shown, 8 of the 17 samples that were re-analyzed by EMSL-Cinnaminson were statistically different from the original analysis performed by EMSL-Libby (based on a Poisson ratio comparison test at a 90% confidence interval). Similar to what was observed in the 2012 re-analyses, there appears to be a consistent bias, with concentrations reported by EMSL-Libby tending to be lower than those reported by EMSL-Cinnaminson. However, concentrations in most samples were usually within a factor of about 2.

[3] For two samples, the re-analysis was performed from the original filter because no raw water remained (these samples are indicated in the table).

Of particular interest are the results for samples P5-20325 and P5-20326. These two samples were preferentially selected for re-analysis because the originally reported LA concentrations suggested that the results for the pore water and its paired surface water got mixed up. The re-analysis performed by EMSL-Cinnaminson confirmed that a filter mix up did occur and that it happened in EMSL-Libby when reporting the results (not in the field)[4].

Conclusions

The results of these re-analyses support the conclusion that filter mix-ups occurred at EMSL-Libby both in 2012 and 2013. The largest mix-up appears to be associated with the set of filters that were prepared during Round 3 of the Phase V, Part A (Kootenai) sampling effort (which included P5-10014, P5-10015, P5-10017, and P5-10018). However, other filter mix-ups outside of this timeframe were also noted, and even occurred during the 2013 study after corrective actions were to have been implemented.

The re-analyses also show that there are differences between the EMSL laboratories in the identification and recording of LA structures in water samples from OU3, albeit the magnitude of the differences in the reported water concentrations are not large (usually within a factor of 2-3).

LIBBY OU3: 2012 PHASE V, SURFACE WATER RE-ANALYSIS RESULTS
REPREPARATION RESULT COMPARISON

Investigation	Repreparation Type	Index ID	Original Analysis (2012)				Repreparation Analysis (Jul/Aug 2013, EMSL-Cinnaminson)				Poisson Rate Comparison (90% CI)
			Laboratory	Total LA Structures	Sensitivity (1/L)	Total LA Conc (MFL	Laboratory	Total LA Structures	Sensitivity (1/L)	Total LA Conc (MFL	
2012 Phase V Part A Surface Water	Raw water	P5-10005	EMSL27	2	3.3E+05	**0.7**	EMSL04	0	1.3E+06	**0**	[0-13.62] The rates are not different
	Raw water	P5-10006	EMSL27	121	3.5E+06	**419**	EMSL04	39	1.2E+07	**473**	[0.65-1.23] The rates are not different
	Raw water	P5-10011	EMSL27	0	1.6E+05	**0**	EMSL04	0	6.4E+05	**0**	Both counts are 0; the rates are not different
	Raw water	P5-10012	EMSL27	27	1.5E+06	**42**	EMSL04	25	1.9E+06	**47**	[0.54-1.46] The rates are not different
	Filter	**P5-10015**	**EMSL27**	**26**	**2.3E+06**	**60**	**EMSL04**	**0**	**1.8E+05**	**0**	**[0-0.01] Rate 1 is greater than Rate 2**
	Raw water	**P5-10017**	**EMSL27**	**25**	**2.3E+06**	**58**	**EMSL27**	**1**	**6.4E+05**	**0.6**	**[17.65-1828.91] Rate 1 is greater than Rate 2**
	Raw water	**P5-10018**	**EMSL27**	**25**	**1.4E+06**	**35**	**EMSL04**	**0**	**6.4E+05**	**0**	**[0-0.06] Rate 1 is greater than Rate 2**
	Raw water	P5-10025	EMSL27	27	2.8E+06	**75**	EMSL04	26	1.9E+06	**51**	[0.91-2.42] The rates are not different
	Raw water	P5-10033	EMSL22	1	4.9E+04	**0.05**	EMSL04	3	2.8E+04	**0.09**	[0.02-5.24] The rates are not different
	Raw water	P5-10034	EMSL22	121	2.8E+05	**33**	EMSL04	114	2.7E+05	**31**	[0.87-1.36] The rates are not different
	Raw water	P5-10053	EMSL04	0	5.0E+04	**0**	EMSL04	1	2.1E+04	**0.02**	[0-44.2] The rates are not different
	Raw water	P5-10056	EMSL04	66	2.5E+05	**16**	EMSL04	84	2.4E+05	**20**	[0.6-1.05] The rates are not different
2012 Phase V Part B Eyed Egg Surface Water	Raw water	P5-20002	EMSL27	58	6.9E+05	**40**	EMSL04	26	1.6E+06	**42**	[0.63-1.46] The rates are not different
	Raw water	P5-20006	EMSL27	33	6.9E+05	**23**	EMSL04	26	8.1E+05	**21**	[0.68-1.74] The rates are not different
	Raw water	P5-20011	EMSL27	25	7.9E+04	**2**	EMSL04	25	2.6E+05	**7**	[0.18-0.5] Rate 1 is less than Rate 2
2012 Phase V Part B Fry Surface Water	Raw water	P5-20016	EMSL04	46	8.2E+05	**38**	EMSL04	60	8.2E+05	**49**	[0.54-1.08] The rates are not different
	Filter	P5-20018	EMSL04	0	8.5E+04	**0**	EMSL04	0	8.6E+04	**0**	Both counts are 0; the rates are not different
	Raw water	P5-20021	EMSL04	26	5.0E+05	**13**	EMSL04	25	5.5E+05	**14**	[0.58-1.58] The rates are not different
	Raw water	P5-20027	EMSL04	25	6.7E+04	**2**	EMSL04	60	2.4E+06	**146**	[0.01-0.02] Rate 1 is less than Rate 2
	Raw water	P5-20031	EMSL04	41	2.5E+05	**10**	EMSL04	73	2.4E+05	**18**	[0.4-0.79] Rate 1 is less than Rate 2
	Raw water	P5-20042	EMSL22	34	1.0E+06	**34**	EMSL04	39	9.7E+05	**38**	[0.59-1.35] The rates are not different
	Raw water	P5-20045	EMSL27	2	5.2E+04	**0.1**	EMSL04	3	5.1E+04	**0.2**	[0.08-4.34] The rates are not different
	Raw water	P5-20069	EMSL27	79	2.8E+05	**22**	EMSL04	42	9.7E+05	**41**	[0.39-0.75] Rate 1 is less than Rate 2
	Raw water	P5-20081	EMSL27	25	1.4E+05	**3**	EMSL04	31	7.8E+05	**24**	[0.09-0.23] Rate 1 is less than Rate 2

All filters pass the CHISQ test for filter loading evenness.

Original Analysis > Repreparation Analysis
Original Analysis < Repreparation Analysis

Notes:

LA - Libby amphibole
-- = result not available
L = liter
MFL - million fibers per liter
% = percent
CI = confidence interval
TEM = transmission electron microscopy

Repreparation analysis confirms suspected filter mix-up at the laboratory during the original analysis.

Lab identifier is in error; corrected EDD is pending

[4] As shown in the table, EMSL-Cinnaminson performed an extra repreparation analysis which confirmed their results for sample P5-20326.

LIBBY OU3: PHASE V PART B, 2013 EYED EGG STUDY, WATER SAMPLING RESULTS

REPREPARATION RESULT COMPARISON

Repreparation Type	Media Type	Index ID	Original Analysis (EMSL-Libby)			Repreparation Analysis (EMSL - Cinnaminson)			Poisson Rate Comparison (90% CI)
			Total LA Structures	Sensitivity (1/L)	Total LA Conc (MFL)	Total LA Structures	Sensitivity (1/L)	Total LA Conc (MFL)	
Reprep from filter	Surface Water	P5-20290	27	1.4E+06	**38**	25	9.8E+05	**25**	[0.96-2.57] The rates are not different
	Surface Water	P5-20294	27	1.2E+06	**34**	25	8.9E+05	**22**	[0.92-2.48] The rates are not different
	Pore Water	P5-20299	28	2.5E+06	**70**	50	2.5E+06	**123**	[0.37-0.86] Rate 1 is less than Rate 2
	Surface Water	P5-20300	0	1.2E+05	**0**	6	1.3E+05	**0.8**	[0-0.59] Rate 1 is less than Rate 2
	Surface Water	P5-20309	26	1.3E+06	**35**	25	2.5E+06	**61**	[0.34-0.93] Rate 1 is less than Rate 2
	Pore Water	P5-20336	1	8.3E+04	**0.08**	2	8.6E+04	**0.2**	[0.02-6.16] The rates are not different
	Pore Water	P5-20324	27	1.3E+06	**36**	37	1.3E+06	**48**	[0.47-1.16] The rates are not different
	Surface Water	**P5-20325**	**26**	**1.7E+06**	**43**	**25**	**1.1E+05**	**2.6**	**[9.93-27.01] Rate 1 is greater than Rate 2**
	Pore Water	**P5-20326**	**0**	**7.8E+04**	**0**	**33**	**1.6E+06**	**54**	**[0-0] Rate 1 is less than Rate 2**
						46	**1.6E+06**	**75**	**[0-0] Rate 1 is less than Rate 2 ****
	Surface Water	P5-20331	25	3.7E+05	**9**	25	6.5E+05	**16**	[0.34-0.94] Rate 1 is less than Rate 2
Reprep from water	Pore Water	P5-20338	34	1.7E+06	**56**	32	1.3E+06	**41**	[0.88-2.11] The rates are not different
	Surface Water	P5-20341	25	2.4E+05	**6**	25	2.5E+05	**6**	[0.57-1.58] The rates are not different
	Pore Water	P5-20348	30	1.7E+06	**50**	32	9.2E+05	**30**	[1.07-2.64] Rate 1 is greater than Rate 2
	Surface Water	P5-20356	25	7.1E+05	**18**	31	1.1E+06	**33**	[0.33-0.86] Rate 1 is less than Rate 2
	Pore Water	P5-20352	0	8.3E+04	**0**	3	8.5E+04	**0.3**	[0-1.67] The rates are not different
	Pore Water	P5-20363	0	1.3E+05	**0**	0	1.3E+05	**0**	Both counts are 0; the rates are not different
	Surface Water	P5-20369	0	1.2E+05	**0**	0	1.3E+05	**0**	Both counts are 0; the rates are not different

All filters pass the CHISQ test for filter loading evenness.

Original Analysis > Repreparation Analysis

Notes:

Original Analysis < Repreparation Analysis

LA - Libby amphibole

-- = result not available

L = liter

MFL - million fibers per liter

% = percent

CI = confidence interval

TEM = transmission electron microscopy

These samples were selected for repreparation analysis by EMSL-Cinnaminson because it was suspected that the paired pore water and surface water results were mixed up by EMSL-Libby. The filter repreparation results confirm that the results were reported incorrectly by EMSL-Libby.

**EMSL-Cinnaminson performed a second repreparation for this filter which confirmed the first repreparation.